

Supporting Information

Palladium-Catalyzed α -Vinylolation of Carbonyl Compounds

Jinkun Huang,* Emilio Bunel, Margaret Faul

*Chemistry Research and Discovery, Amgen Inc., One Amgen Center
Drive, Thousand Oaks, California 91 320*

Table of Contents:

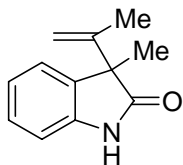
Experimental Section (procedures and characterization data)	Pages 2-10
Copies of NMR (^1H , ^{13}C , ^{19}F) spectra	Pages 11-53

EXPERIMENTAL

General. All anhydrous, sure-seal grade organic solvents including toluene, xylene, tetrahydrofuran (THF), dioxane, 1,2-dimethoxyethane (DME) and N,N-dimethylformamide (DMF) were used as purchased. All commercially available reagents were purchased from commercial sources and used as received. 1,2-dihydro-3-naphthyl tosylate was synthesized according to literature procedures.¹ All reactions were carried out in oven dried glassware under an atmosphere of nitrogen. Purification by flash column chromatography was performed using commercially available pre-packed silica gel plugs and hexanes/ethylacetate solvents. Isolated yields correspond to products of > 95 % purity as determined by GC, LC and NMR. LC yields correspond to calibrated LC yields using an authentic sample as standard.

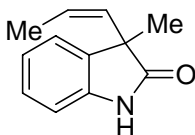
General Procedure for the Vinylation Reactions. To a mixture of 3-methyl oxindole (or ketone and ester, 2.0 mmol), [Pd(P^tBu₃)Br]₂ (0.025 mmol) in toluene (4 mL) was added LHMDS (5.0 mmol). The mixture was stirred for 5 min at room temperature and followed by addition of the corresponding vinyl bromide (or triflate and tosylate, 3 mmol) in one portion. The resulting suspension was stirred at 80 °C for 24 h (48 h for the vinyl tosylate). The reaction mixture was cooled to room temperature and diluted with diethyl ether (10 mL). The ether solution was washed with saturated ammonium chloride (2 x 5 mL), water (5 mL), dried over anhydrous magnesium sulfate and concentrated under vacuum. The crude residue was purified by flash column chromatography on silica gel using hexane/ethylacetate as eluents.

(1) Klapars, A.; Campos, K. R.; Chen, C.-Y.; Volante, R. P. *Org. Lett.* **2005**, 7, 1185.



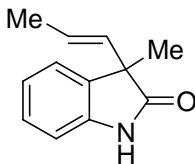
3-methyl-3-(prop-1-en-2-yl)indolin-2-one (Table 2, entry 1)

This compound was isolated as white solid from the reaction of 3-methyl oxindole and 2-bromoprop-1-ene. ^1H NMR (CDCl_3): δ 8.41 (s, 1 H), 7.23 (t, $J = 8.0$ Hz, 1 H), 7.09-7.05 (m, 2 H), 6.94 (d, $J = 8.0$ Hz, 1 H), 5.15 (s, 1 H), 5.10 (s, 1 H), 1.58 (s, 3 H), 1.55 (s, 3 H) ppm; ^{13}C (CDCl_3): δ 181.45, 143.43, 140.45, 134.41, 127.98, 123.48, 122.69, 113.21, 109.80, 54.40, 21.48, 19.54 ppm.



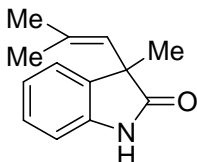
(Z)-3-methyl-3-(prop-1-en-1-yl)indolin-2-one (Table 2, entry 2)

This compound was isolated as white solid from the reaction of 3-methyl oxindole and (Z)-1-bromoprop-1-ene. ^1H NMR (CDCl_3): δ 8.85 (s, 1 H), 7.19 (t, $J = 8.0$ Hz, 1 H), 7.15 (d, $J = 8.0$ Hz, 1 H), 7.03 (t, $J = 8.0$ Hz, 1 H), 6.94 (d, $J = 8.0$ Hz, 1 H), 5.65-5.58 (m, 2 H), 1.56 (s, 3 H), 1.18 (d, $J = 8.0$ Hz, 3 H) ppm; ^{13}C (CDCl_3): δ 182.61, 139.86, 136.13, 130.97, 129.42, 127.62, 123.40, 122.78, 109.92, 49.17, 26.82, 13.54 ppm.



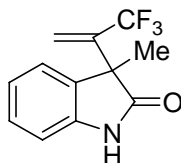
(E)-3-methyl-3-(prop-1-en-1-yl)indolin-2-one (Table 2, entry 3)

This compound was isolated as white solid from the reaction of 3-methyl oxindole and (E)-1-bromoprop-1-ene. ^1H NMR (CDCl_3): δ 8.52 (s, 1 H), 7.22 (t, $J = 8.0$ Hz, 1 H), 7.17 (d, $J = 8.0$ Hz, 1 H), 7.07 (t, $J = 8.0$ Hz, 1 H), 6.95 (d, $J = 8.0$ Hz, 1 H), 5.64-5.56 (m, 2 H), 1.56 (s, 3 H), 1.18 (d, $J = 4.0$ Hz, 3 H) ppm; ^{13}C (CDCl_3): δ 181.83, 140.07, 134.11, 130.85, 127.85, 126.40, 124.12, 122.43, 109.93, 50.92, 22.89, 17.94 ppm.



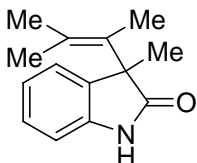
3-methyl-3-(2-methylprop-1-en-1-yl)indolin-2-one (Table 2, entry 4)

This compound was isolated as white solid from the reaction of 3-methyl oxindole and 1-bromo-2-methylprop-1-ene. ^1H NMR (CDCl_3): δ 8.83 (s, 1 H), 7.19 (t, $J = 8.0$ Hz, 1 H), 7.13 (d, $J = 8.0$ Hz, 1 H), 7.03 (t, $J = 8.0$ Hz, 1 H), 6.94 (d, $J = 8.0$ Hz, 1 H), 5.43 (s, 1 H), 1.74 (s, 3 H), 1.53 (s, 3 H), 1.16 (s, 3 H) ppm; ^{13}C (CDCl_3): δ 183.08, 139.82, 137.81, 136.66, 127.41, 125.19, 123.30, 122.72, 109.83, 49.12, 27.55, 26.67, 18.30 ppm.



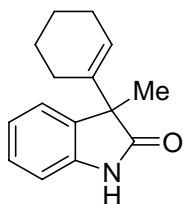
3-methyl-3-(1,1,1-trifluoroprop-2-en-2-yl)indolin-2-one (Table 2, entry 5)

This compound was isolated as white solid from the reaction of 3-methyl oxindole and 2-bromo-3,3,3-trifluoroprop-1-ene. ^1H NMR (CDCl_3): δ 8.95 (s, 1 H), 7.16 (t, $J = 8.0$ Hz, 1 H), 7.03 (d, $J = 8.0$ Hz, 1 H), 6.96 (t, $J = 8.0$ Hz, 1 H), 6.88 (d, $J = 8.0$ Hz, 1 H), 6.06 (s, 1 H), 5.86 (s, 1 H), 1.54 (s, 3 H) ppm; ^{13}C (CDCl_3): δ 179.86, 140.19, 138.36 (q, $J = 28$ Hz), 132.12, 128.66, 124.42, 123.87, 122.74, 121.68, 110.43, 50.50, 23.37 ppm; ^{19}F (CDCl_3): δ -62.73 ppm.



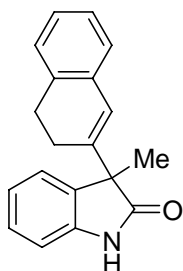
3-methyl-3-(3-methylbut-2-en-2-yl)indolin-2-one (Table 2, entry 6)

This compound was isolated as white solid from the reaction of 3-methyl oxindole and 2-bromo-3-methylbut-2-ene. ^1H NMR (CDCl_3): δ 8.69 (s, 1 H), 7.17 (t, $J = 8.0$ Hz, 1 H), 7.11 (d, $J = 8.0$ Hz, 1 H), 7.00 (t, $J = 8.0$ Hz, 1 H), 6.92 (d, $J = 8.0$ Hz, 1 H), 1.97 (s, 3 H), 1.72 (s, 3 H), 1.53, 1.19 (s, 3 H) ppm; ^{13}C (CDCl_3): δ 183.88, 139.32, 137.88, 130.43, 127.21, 125.95, 122.74, 122.58, 109.93, 53.06, 26.46, 23.05, 21.26, 17.57 ppm.



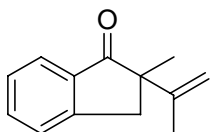
3-cyclohexenyl-3-methylindolin-2-one (Table 2, entry 7)

This compound was isolated as white solid from the reaction of 3-methyl oxindole and 1-cyclohexenyl triflate. ^1H NMR (CDCl_3): δ 9.11 (s, 1 H), 7.03 (t, $J = 8.0$ Hz, 1 H), 6.89 (d, $J = 8.0$ Hz, 1 H), 6.85 (t, $J = 8.0$ Hz, 1 H), 6.79 (d, $J = 8.0$ Hz, 1 H), 5.71 (m, 1 H), 1.97 (m, 2 H), 1.61 (m, 1 H), 1.46-1.27 (m, 8 H) ppm; ^{13}C (CDCl_3): δ 182.93, 140.81, 135.97, 135.20, 127.71, 123.63, 123.30, 122.50, 109.95, 54.47, 25.49, 25.07, 22.75, 22.10, 21.07 ppm.



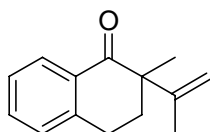
3-(3,4-dihydronaphthalen-2-yl)-3-methylindolin-2-one (Scheme 1)

This compound was isolated as white solid from the reaction of 3-methyl oxindole and 1,2-dihydro-3-naphthyl tosylate. ^1H NMR (CDCl_3): δ 8.73 (s, 1 H), 7.22 (t, $J = 8.0$ Hz, 1 H), 7.15-7.01 (m, 6 H), 6.85 (t, $J = 8.0$ Hz, 1 H), 6.75 (d, $J = 8.0$ Hz, 1 H), 6.59 (s, 1 H), 2.74-2.64 (m, 2 H), 2.10-2.00 (m, 2 H), 1.65 (s, 3 H) ppm; ^{13}C (CDCl_3): δ 181.53, 140.62, 139.06, 134.88, 134.32, 133.97, 128.17, 127.12, 126.45, 124.18, 123.76, 122.79, 109.99, 54.17, 28.19, 24.36, 20.98 ppm.



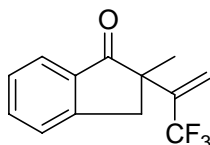
2-methyl-2-(prop-1-en-2-yl)-1-indanone (Table 3, entry 1a)

This compound was isolated as colorless oil from the reaction of 2-methyl-1-indanone and 2-bromoprop-1-ene. ^1H NMR (CDCl_3): δ 7.79 (d, $J = 8.0$ Hz, 1 H), 7.61 (t, $J = 8$ Hz, 1 H), 7.46 (d, $J = 8$ Hz, 1 H), 7.39 (t, $J = 8$ Hz, 1 H), 4.96 (s, 1 H), 4.95 (s, 1 H), 3.34 (d, $J = 20$ Hz, 1 H), 2.96 (d, $J = 20$ Hz, 1 H), 1.65 (s, 3 H), 1.38 (s, 3 H) ppm; ^{13}C (CDCl_3): δ 209.15, 152.70, 145.86, 135.88, 134.98, 127.56, 126.49, 126.40, 112.11, 54.57, 41.31, 22.67, 19.91 ppm.



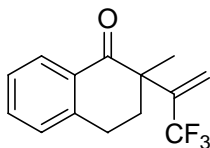
2-methyl-2-(prop-1-en-2-yl)-1-tetralone (Table 3, entry 1b)

This compound was isolated as colorless oil from the reaction of 2-methyl-1-tetralone and 2-bromoprop-1-ene. ^1H NMR (CDCl_3): δ 8.06 (d, $J = 8.0$ Hz, 1 H), 7.43 (t, $J = 8$ Hz, 1 H), 7.29 (t, $J = 8$ Hz, 1 H), 7.18 (d, $J = 8$ Hz, 1 H), 4.87 (s, 1 H), 4.58 (s, 1 H), 2.96 (m, 1 H), 2.83 (m, 1 H), 2.33 (m, 1 H), 1.96 (m, 1 H), 1.80 (s, 3 H), 1.34 (s, 3 H) ppm; ^{13}C (CDCl_3): δ 201.53, 145.42, 143.57, 132.95, 132.46, 128.58, 127.74, 126.56, 113.22, 51.56, 34.25, 26.15, 23.00, 19.49 ppm.



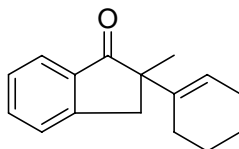
2-methyl-2-(1,1,1-trifluoroprop-2-en-2-yl)-1-indanone (Table 3, entry 2a)

This compound was isolated as colorless oil from the reaction of 2-methyl-1-indanone and 2-bromo-3,3,3-trifluoroprop-1-ene. ^1H NMR (CDCl_3): δ 7.67 (d, $J = 8.0$ Hz, 1 H), 7.50 (t, $J = 8$ Hz, 1 H), 7.31 (t, $J = 8$ Hz, 1 H), 7.26 (d, $J = 8$ Hz, 1 H), 5.82 (s, 1 H), 5.56 (s, 1 H), 3.37 (d, $J = 16$ Hz, 1 H), 2.91 (d, $J = 16$ Hz, 1 H), 1.29 (s, 3H) ppm; ^{13}C (CDCl_3): δ 206.00, 151.49, 140.26 (q, $J = 27$ Hz), 135.36, 134.44, 127.82, 126.63, 125.01, 121.47, 121.41, 51.01, 40.93, 24.03 ppm. ^{19}F (CDCl_3): δ -61.36 ppm.



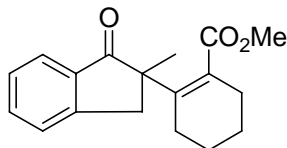
2-methyl-2-(1,1,1-trifluoroprop-2-en-2-yl)-1-tetralone (Table 3, entry 2 b)

This compound was isolated as colorless oil from the reaction of 2-methyl-1-tetralone and 2-bromo-3,3,3-trifluoroprop-1-ene. ^1H NMR (CDCl_3): δ 8.08 (d, $J = 8.0$ Hz, 1 H), 7.48 (t, $J = 8$ Hz, 1 H), 7.33 (t, $J = 8$ Hz, 1 H), 7.23 (d, $J = 8$ Hz, 1 H), 5.90 (s, 1 H), 5.45 (s, 1 H), 2.99 (m, 1 H), 2.54 (m, 1 H), 2.00 (m, 1 H), 1.48 (s, 3 H), 1.27 (m, 1 H) ppm; ^{13}C (CDCl_3): δ 198.44, 142.99, 141.00 (q, $J = 27$ Hz), 133.55, 131.48, 128.72, 128.33, 126.93, 125.01, 121.87, 49.24, 33.87, 25.78, 21.87 ppm. ^{19}F (CDCl_3): δ -59.92 ppm.



2-cyclohexenyl-2-methyl-1-indanone (Table 3, entry 3a)

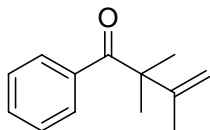
This compound was isolated as colorless oil from the reaction of 2-methyl-1-indanone and 1-cyclohexenyl triflate. ^1H NMR (CDCl_3): δ 7.78 (d, $J = 8.0$ Hz, 1 H), 7.60 (t, $J = 8$ Hz, 1 H), 7.44 (d, $J = 8$ Hz, 1 H), 7.38 (t, $J = 8$ Hz, 1 H), 5.66 (m, 1 H), 3.31 (d, $J = 16$ Hz, 1 H), 2.93 (d, $J = 16$ Hz, 1 H), 2.07 (m, 2 H), 1.89 (m, 1 H), 1.76 (m, 1 H), 1.57 (m, 4 H), 1.34 (s, 3 H) ppm; ^{13}C (CDCl_3): δ 210.04, 152.94, 138.34, 135.56, 134.79, 127.39, 126.41, 124.42, 122.26, 54.44, 41.59, 25.42, 23.02, 22.18 ppm.



methyl 2-(2-methyl-1-indanon-yl)cyclohex-1-enecarboxylate (Table 3, entry 3b)

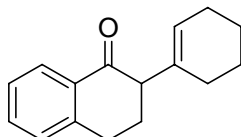
This compound was isolated as colorless oil from the reaction of 2-methyl-1-indanone and 2-methylcarboxyl-1-cyclohexenyl triflate. ^1H NMR (CDCl_3): δ 7.77 (d, $J = 8.0$ Hz, 1 H), 7.58 (t, $J = 8$ Hz, 1 H), 7.41 (d, $J = 8$ Hz, 1 H), 7.38 (t, $J = 8$ Hz, 1 H), 3.35 (d, $J = 16$ Hz, 1 H), 3.19 (s, 3 H), 3.05 (d, $J = 16$ Hz, 1 H), 2.58 (m, 1 H), 2.27 (m, 2 H), 2.10 (m, 1 H), 1.82-1.73 (m, 2 H), 1.53 (m, 2 H), 1.28 (s, 3 H) ppm; ^{13}C (CDCl_3): δ 207.38,

170.36, 150.35, 144.12, 136.07, 134.35, 127.23, 126.55, 124.43, 54.27, 50.81, 43.86, 28.65, 27.88, 23.60, 22.70, 21.60 ppm.



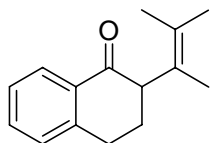
2,2,3-trimethyl-1-phenylbut-3-en-1-one (Table 3, entry 4)

This compound was isolated as colorless oil from the reaction of 2-methyl-1-phenylpropan-1-one and 2-bromoprop-1-ene. ^1H NMR (CDCl_3): δ 7.99 (d, J = 8.0 Hz, 2 H), 7.48 (t, J = 4 Hz, 1 H), 7.37 (t, J = 8 Hz, 2 H), 5.11 (s, 1 H), 5.02 (s, 1 H), 1.75 (s, 3 H), 1.40 (s, 6 H) ppm; ^{13}C (CDCl_3): δ 204.07, 149.71, 136.84, 132.09, 129.01, 128.09, 110.70, 52.89, 26.01, 20.54 ppm.



2-cyclohexenyl-1-tetralone (Table 3, entry 5)

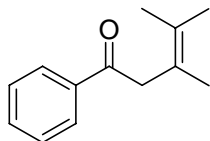
This compound was isolated as colorless oil from the reaction of 1-tetralone and 1-cyclohexenyl triflate. ^1H NMR (CDCl_3): δ 8.04 (d, J = 8.0 Hz, 1 H), 7.45 (t, J = 8 Hz, 1 H), 7.30 (t, J = 8 Hz, 1 H), 7.23 (d, J = 8.0 Hz, 1 H), 5.45 (s, 1 H), 3.11 (dd, J = 8 Hz, J = 4 Hz, 1 H), 2.99 (m, 2 H), 2.25 (m, 1 H), 2.14 (m, 1 H), 2.00 (m, 4 H), 1.65 (m, 4 H) ppm; ^{13}C (CDCl_3): δ 199.17, 144.14, 135.92, 133.16, 133.00, 128.66, 127.43, 126.59, 124.76, 56.16, 28.49, 28.20, 26.80, 25.35, 22.70, 22.35 ppm.



2-(3-methylbut-2-en-2-yl)-1-tetralone (Table 3, entry 6)

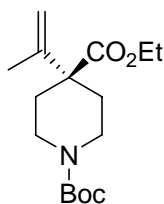
This compound was isolated as a colorless oil from the reaction of 1-tetralone and 2-bromo-3-methylbut-1-ene. ^1H NMR (CDCl_3): δ 8.06 (d, J = 8.0 Hz, 1 H), 7.46 (t, J = 8 Hz, 1 H), 7.31 (t, J = 8 Hz, 1 H), 7.24 (d, J = 8.0 Hz, 1 H), 3.63 (dd, J = 4 Hz, 1 H), 3.09 (m, 1 H), 2.98 (m, 1 H), 2.23 (m, 1 H), 2.05 (m, 1 H), 1.75 (s, 3 H), 1.71 (s, 3 H), 1.60 (s,

3 H) ppm; ^{13}C (CDCl_3): δ 198.95, 144.15, 133.16, 133.14, 128.61, 127.86, 127.52, 126.60, 126.09, 52.66, 29.73, 28.64, 20.83, 20.53, 15.62 ppm.



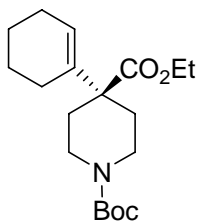
3,4-dimethyl-1-phenylpent-3-en-1-one (Table 3, entry 7)

This compound was isolated as a colorless oil from the reaction of acetophenone and 2-bromo-3-methylbut-1-ene. ^1H NMR (CDCl_3): δ 7.97 (d, J = 8.0 Hz, 1 H), 7.56 (t, J = 4 Hz, 1 H), 7.45 (t, J = 8 Hz, 2 H), 3.75 (s, 2 H), 1.74 (s, 3 H), 1.70 (s, 6 H) ppm; ^{13}C (CDCl_3): δ 198.84, 137.32, 132.89, 128.54, 128.12, 121.48, 44.53, 20.70, 19.25 ppm.



1-tert-butyl 4-ethyl 4-(prop-1-en-2-yl)piperidine-1,4-dicarboxylate (Table 3, entry 8)

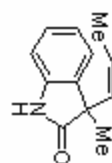
This compound was isolated as a colorless oil from the reaction of 1-tert-butyl 4-ethyl piperidine-1,4-dicarboxylate and 2-bromoprop-1-ene. ^1H NMR (CDCl_3): δ 4.98 (s, 1 H), 4.94 (s, 1 H), 4.20 (q, J = 8 Hz, 2 H), 3.94 (m, 2 H), 2.94 (m, 2 H), 2.25 (m, 2 H), 1.76 (s, 3 H), 1.62 (m, 2 H), 1.47 (s, 9 H), 1.28 (t, J = 8 Hz, 3 H) ppm; ^{13}C (CDCl_3): δ 173.72, 145.73, 112.43, 79.48, 60.89, 50.80, 41.39, 32.09, 28.47, 19.71, 14.20 ppm.



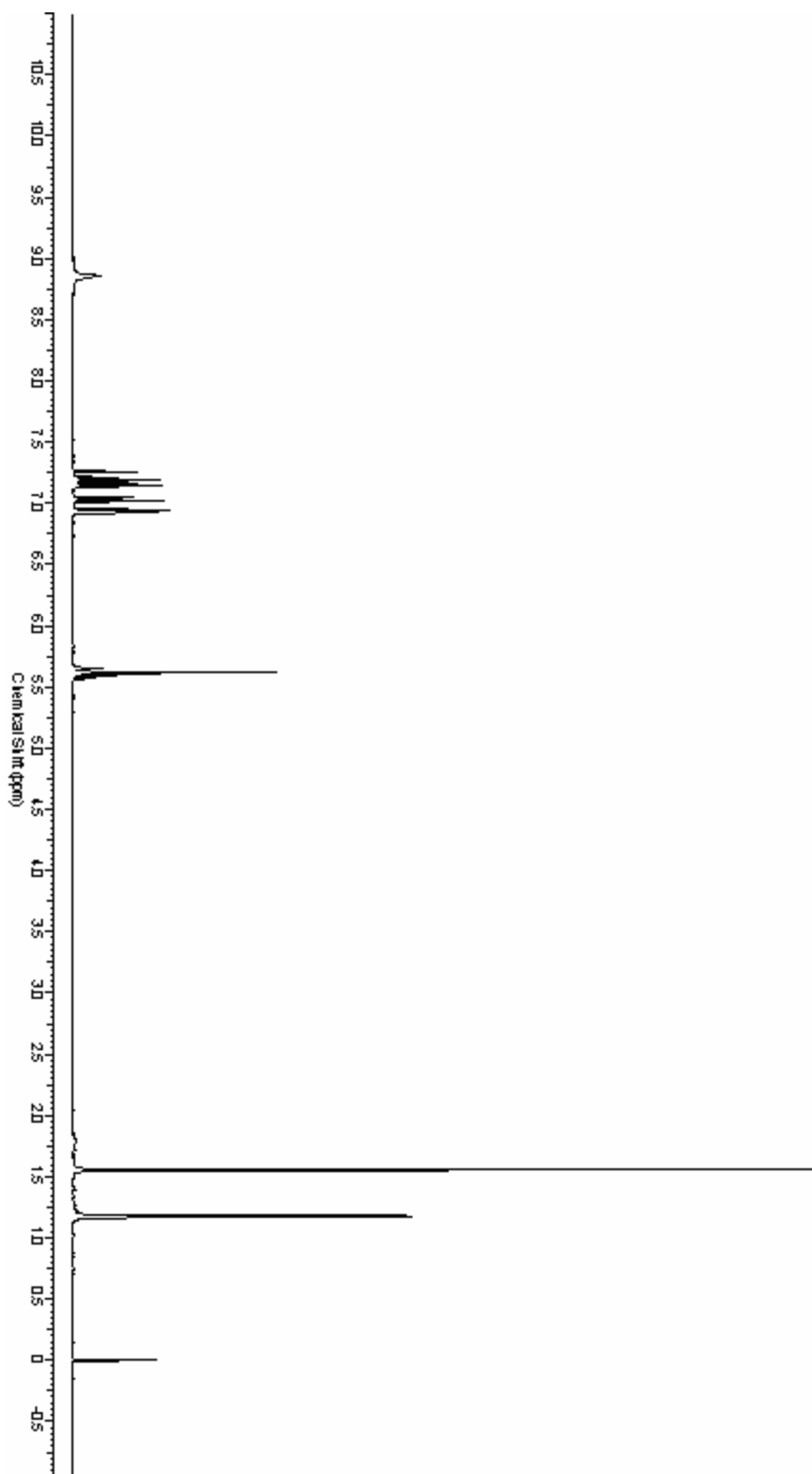
1-tert-butyl 4-ethyl 4-cyclohexenylpiperidine-1,4-dicarboxylate (Table 3, entry 9)

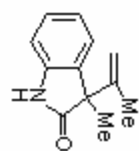
This compound was isolated as a colorless oil from the reaction of 1-tert-butyl 4-ethyl piperidine-1,4-dicarboxylate and 1-cyclohexenyl triflate. ^1H NMR (CDCl_3): δ 5.62 (m, 1 H), 4.16 (q, J = 8 Hz, 2 H), 3.86 (m, 2 H), 2.91 (m, 2 H), 2.17 (m, 2 H), 2.06 (m, 2 H), 1.93 (m, 2 H), 1.60-1.52 (m, 5 H), 1.45 (s, 9 H), 1.24 (t, 3 H) ppm; ^{13}C (CDCl_3): δ

174.25, 154.87, 137.81, 123.06, 79.36, 60.63, 50.68, 41.44, 31.86, 28.49, 25.59, 24.83,
23.10, 22.15, 14.28 ppm.

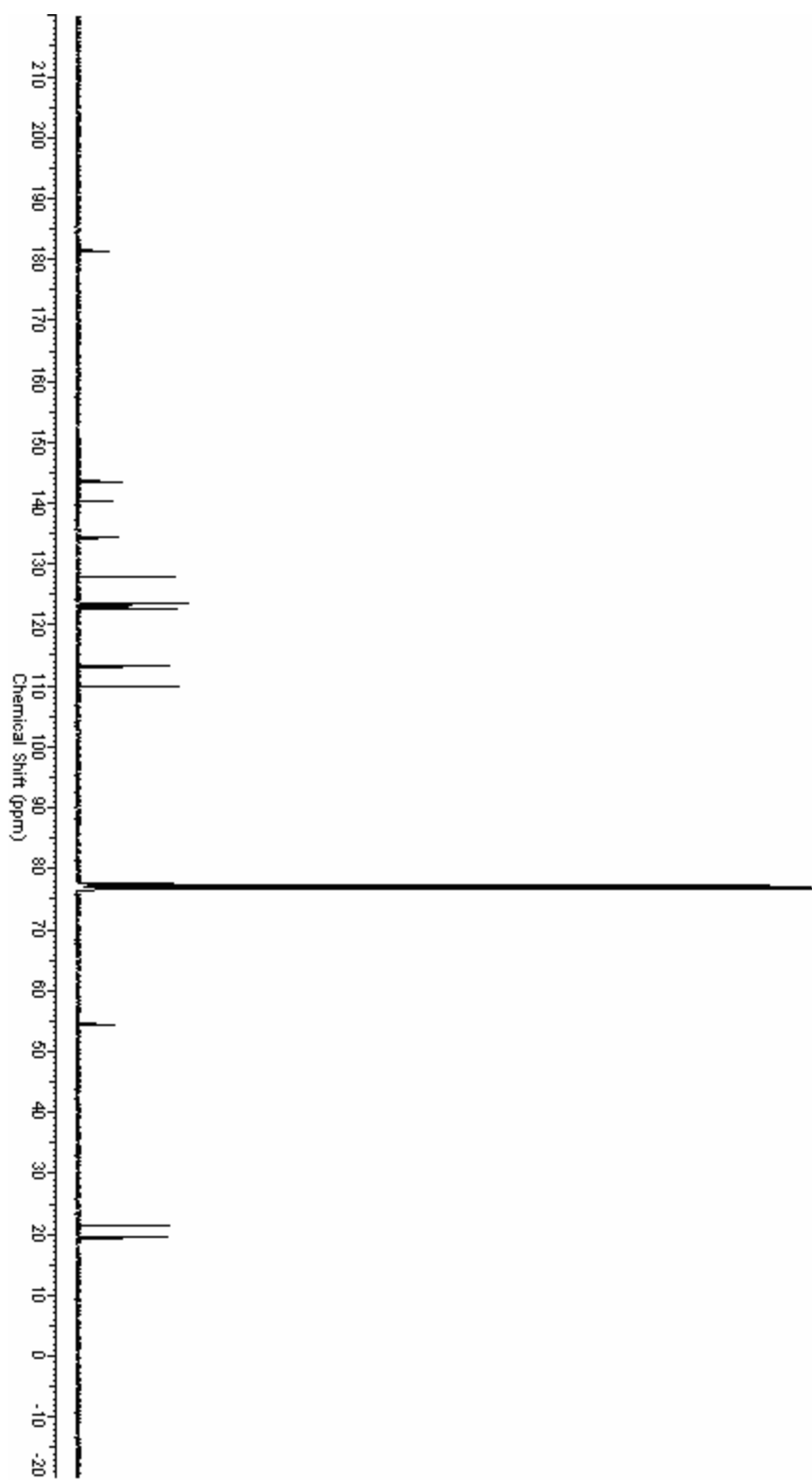


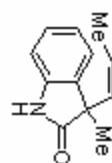
¹H (Table 2, entry 2)



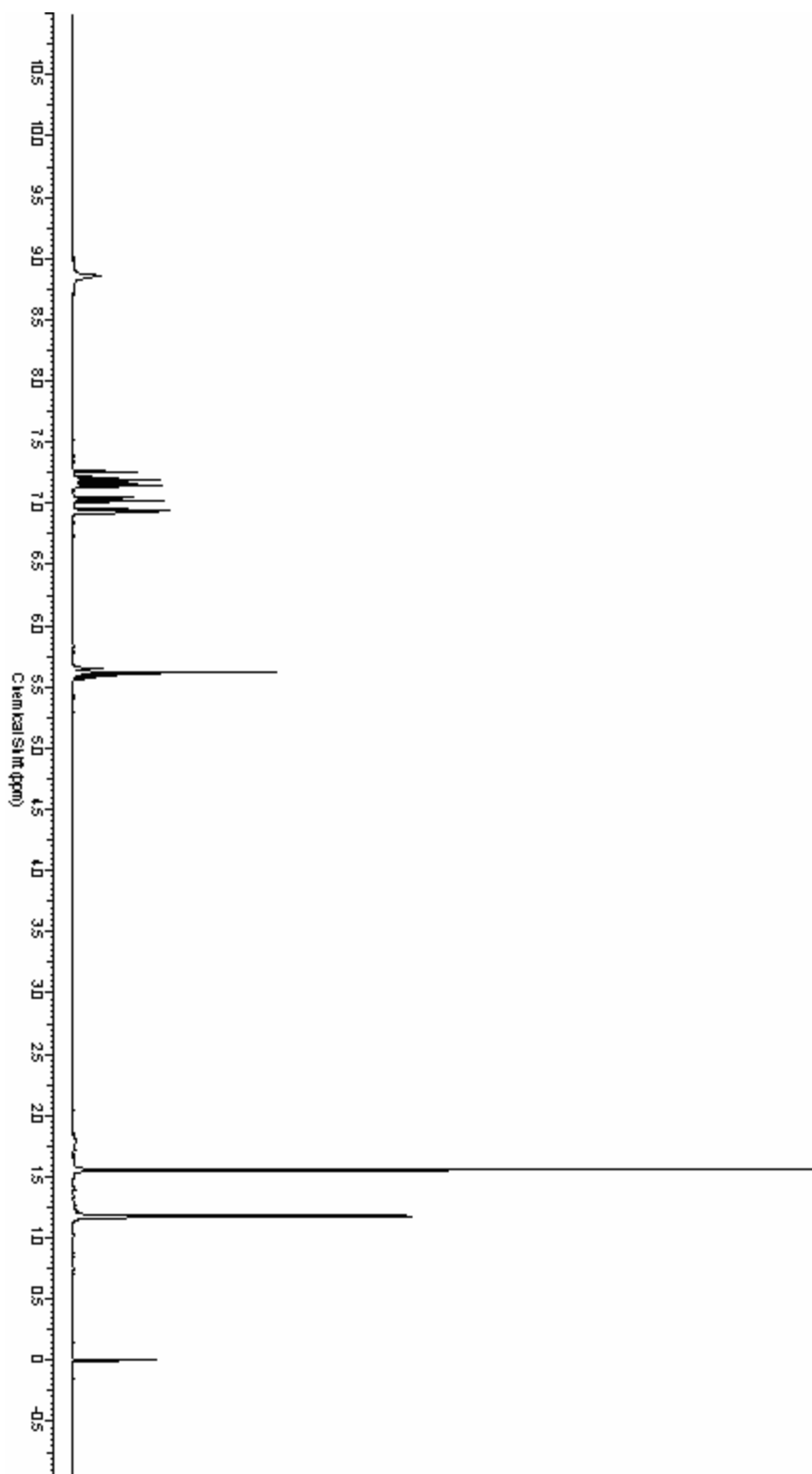


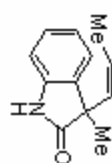
¹³C (Table 2, entry 1)



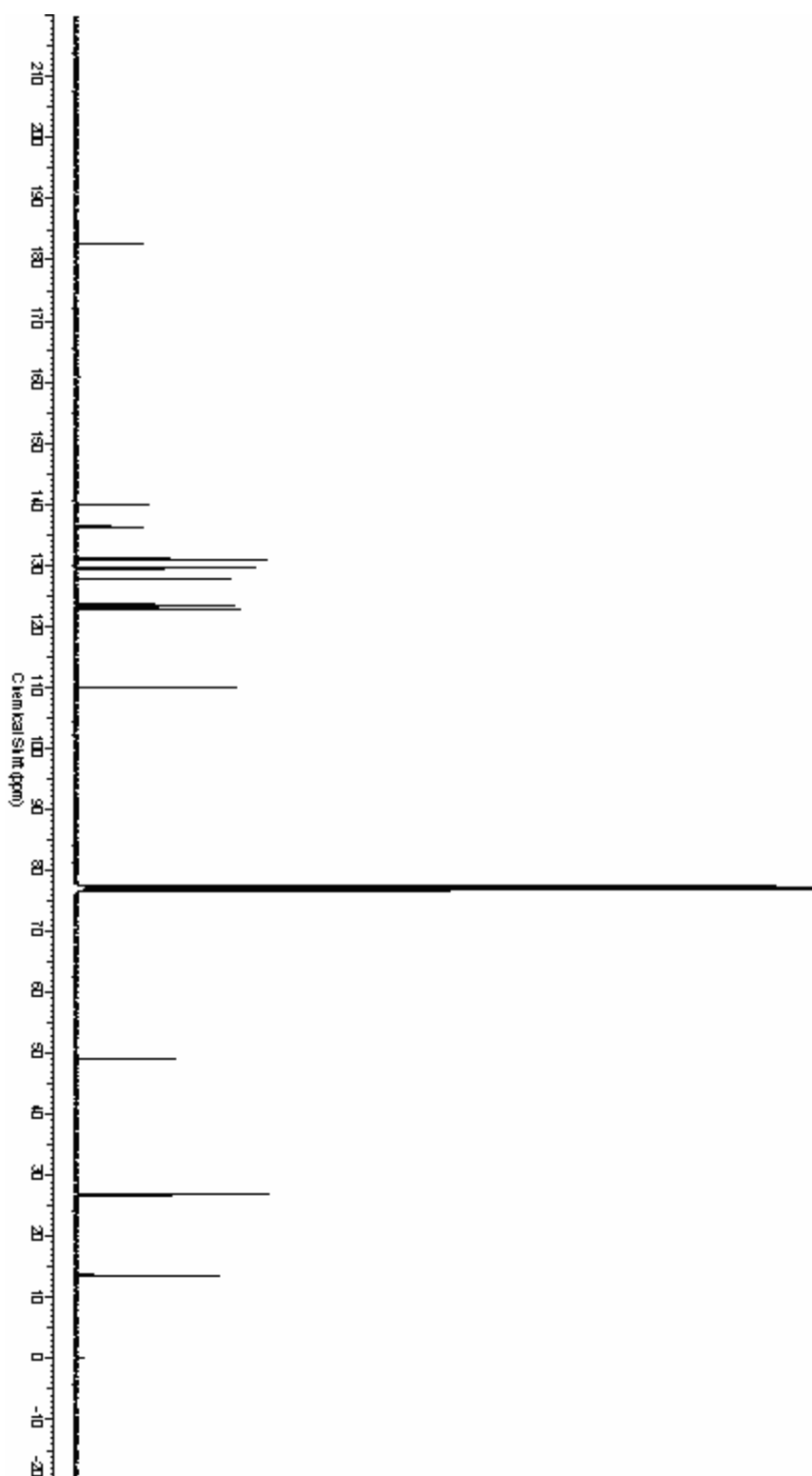


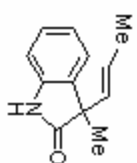
¹H (Table 2, entry 2)



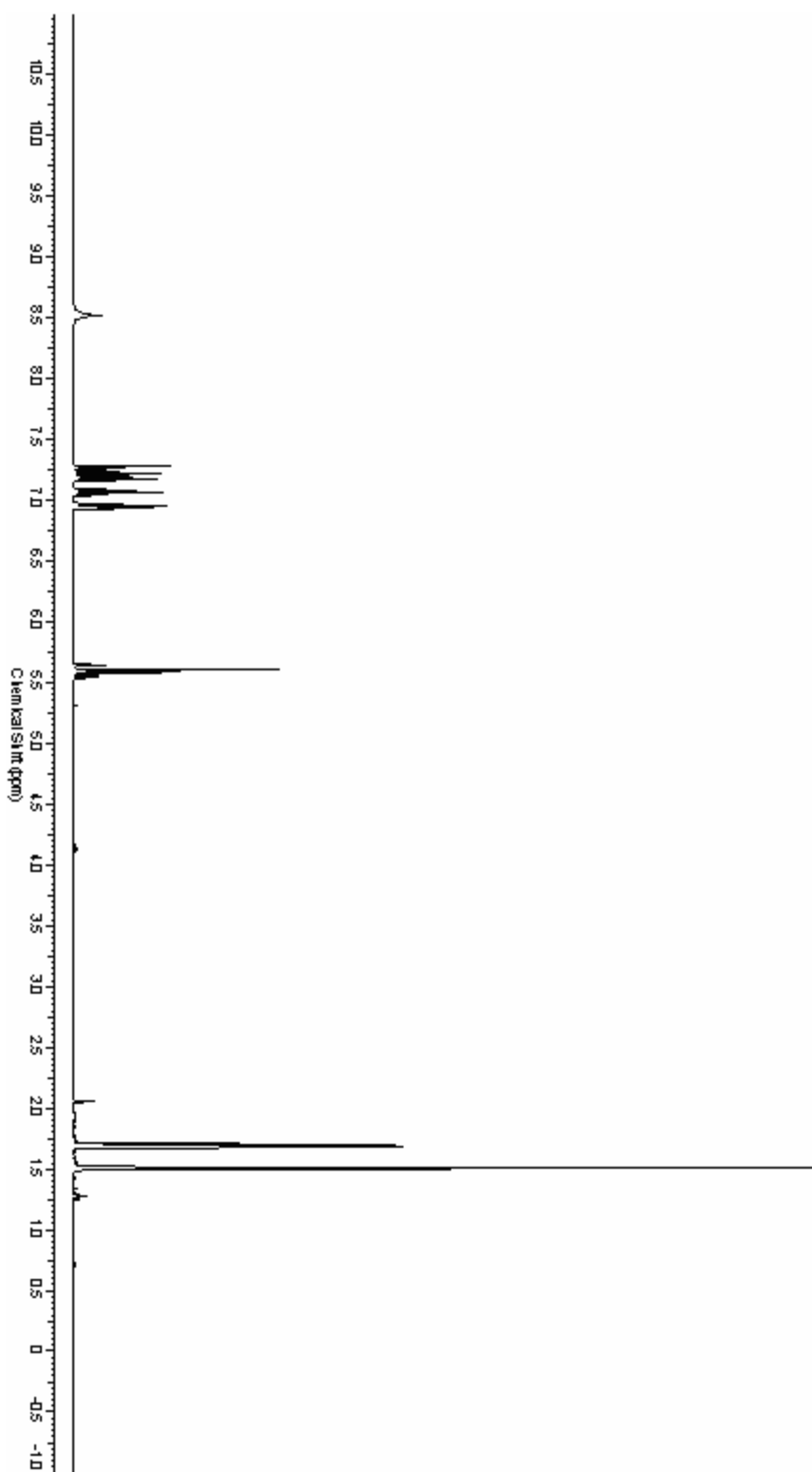


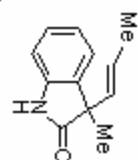
¹³C (Table 2, entry 2)



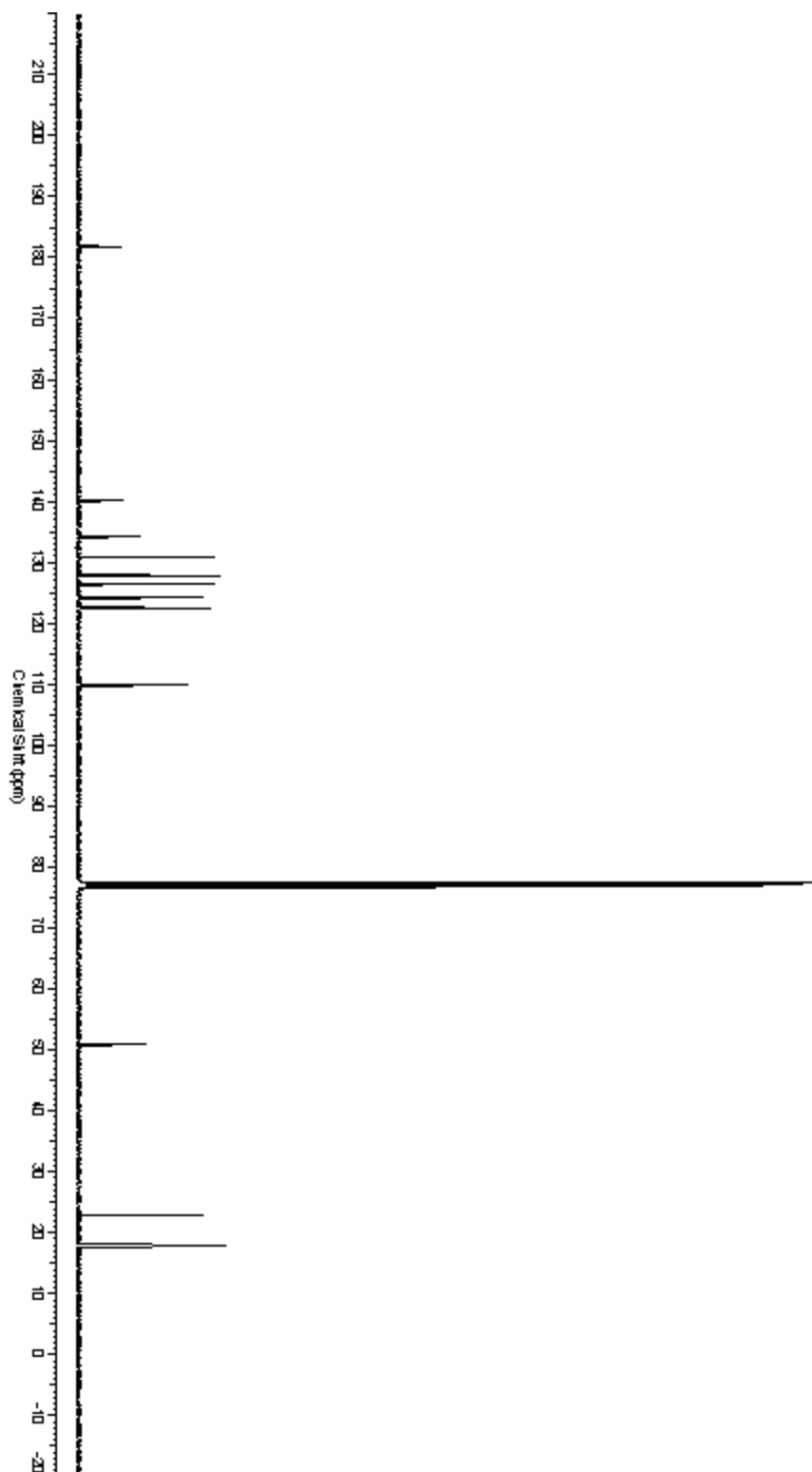


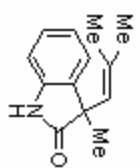
¹H (Table 2, entry 3)



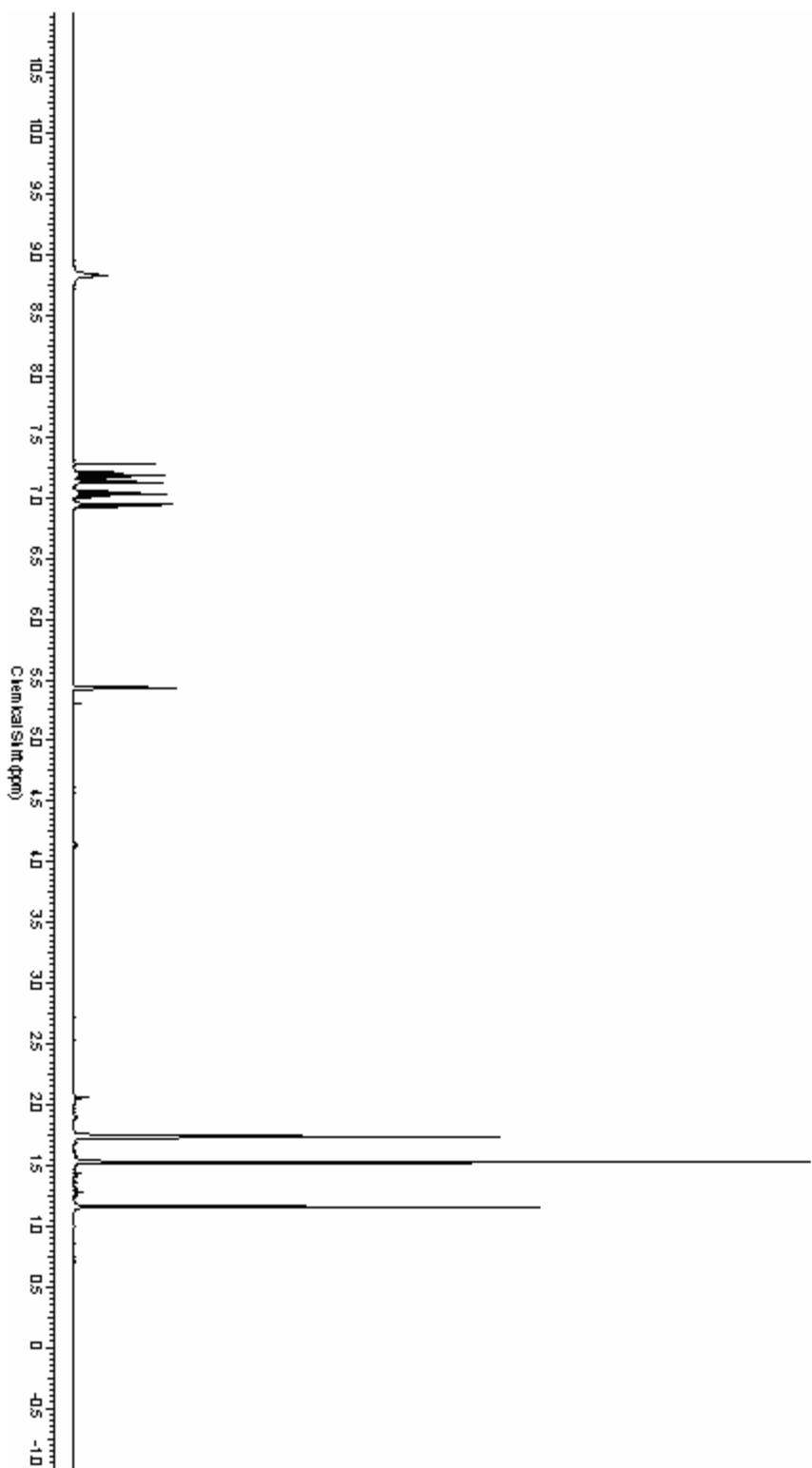


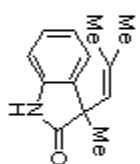
^{13}C (Table 2, entry 3)



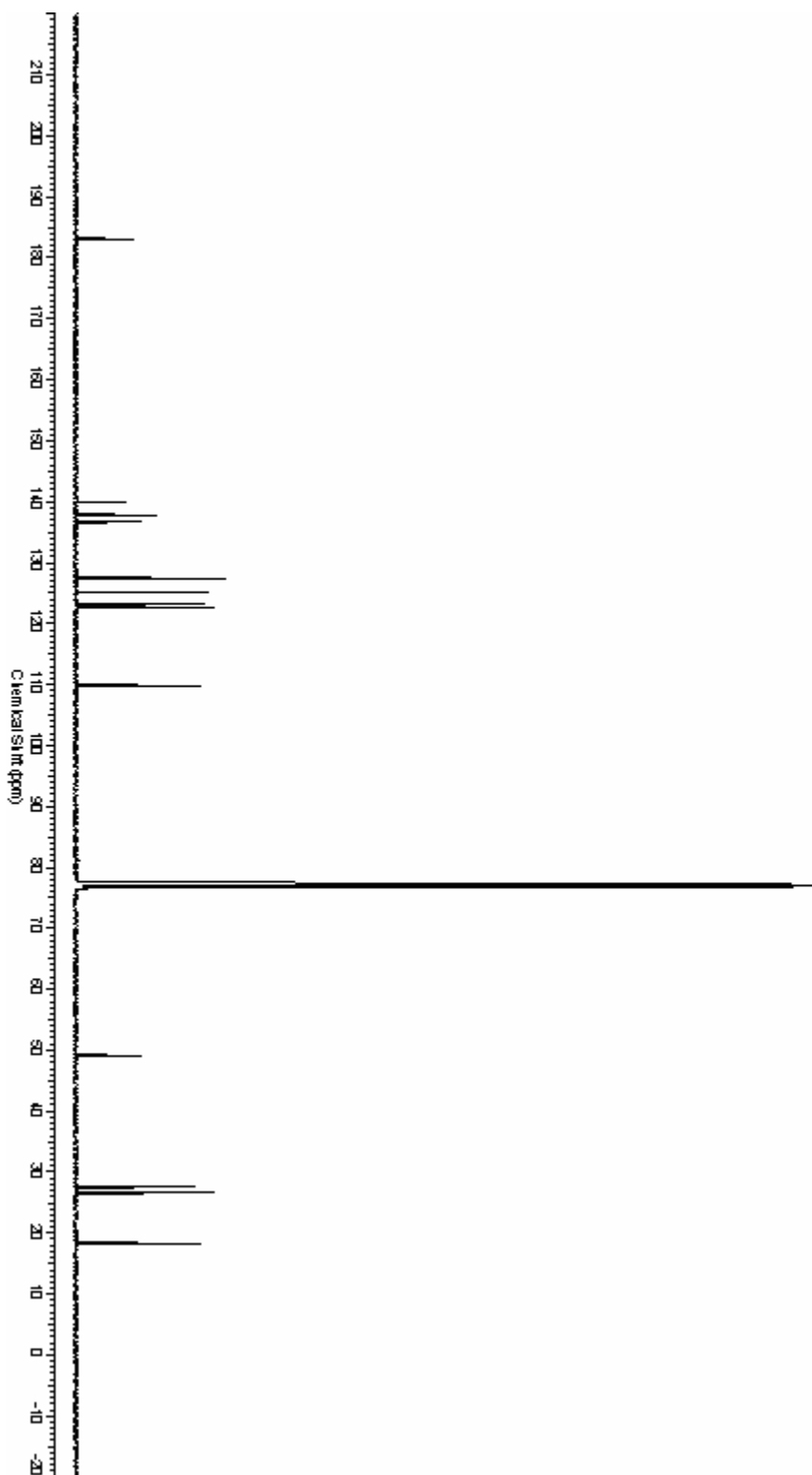


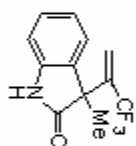
¹H (Table 2, entry 4)



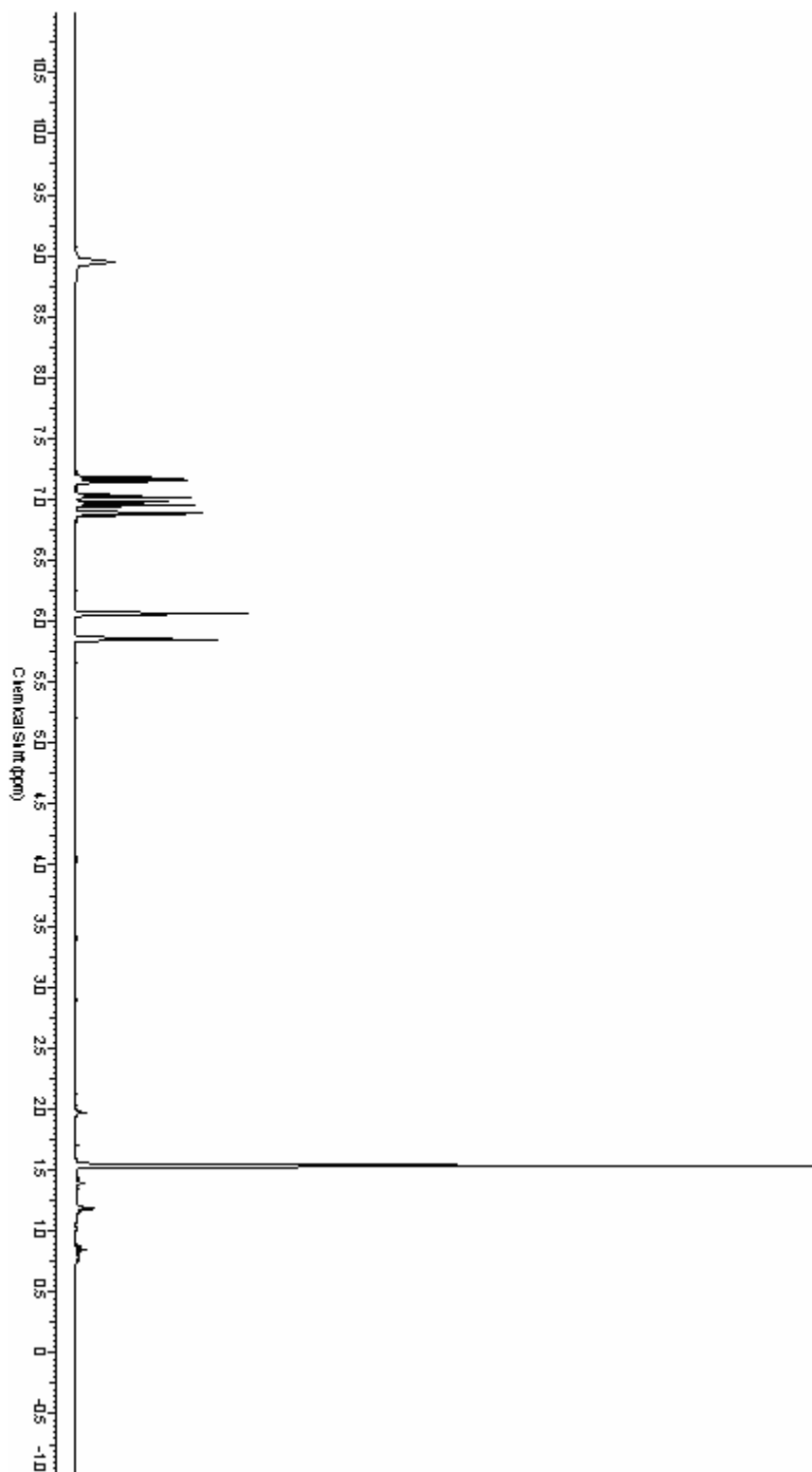


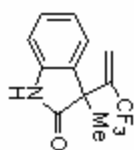
¹³C (Table 2, entry 4)



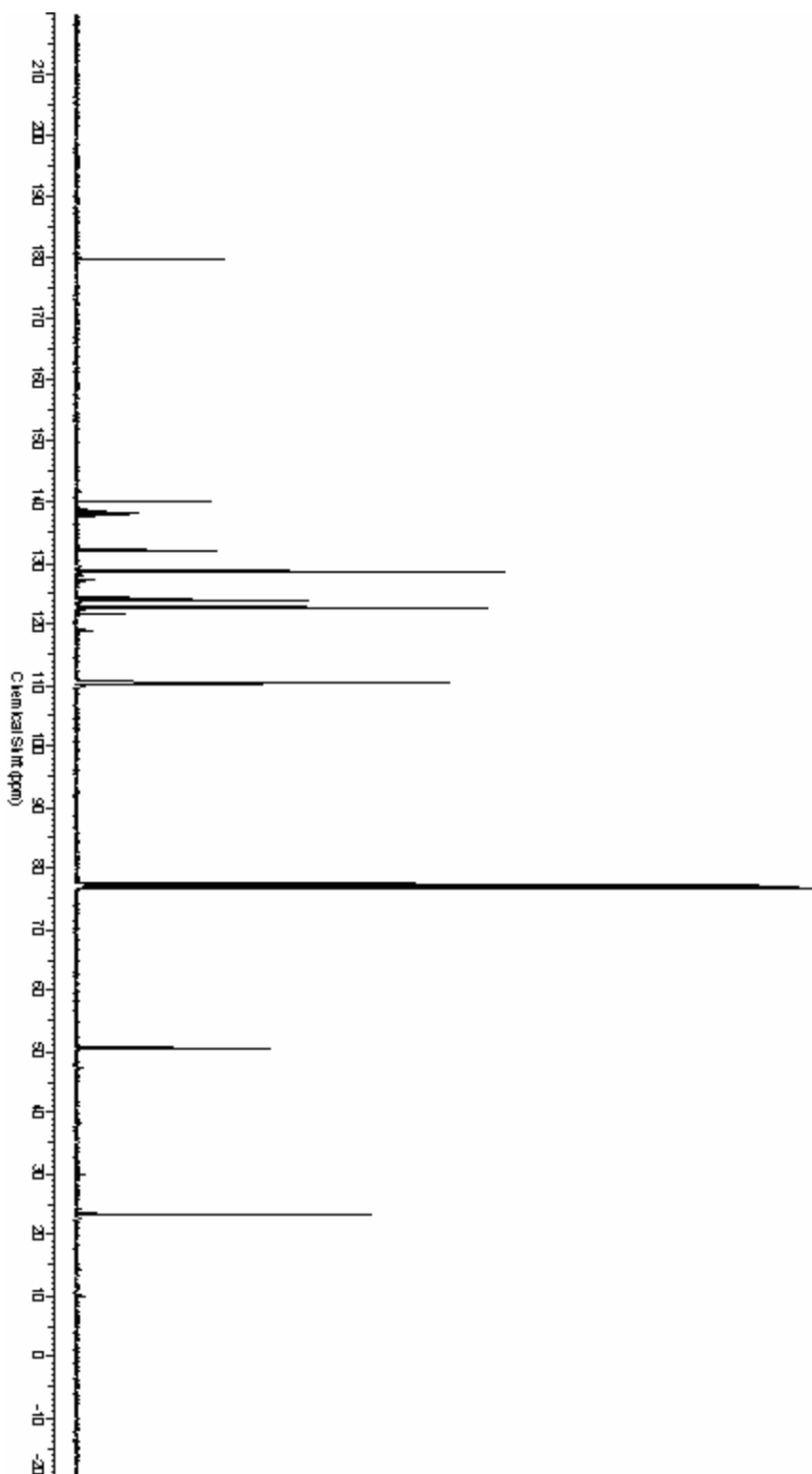


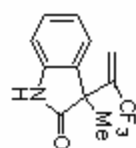
¹H (Table 2, entry 5)



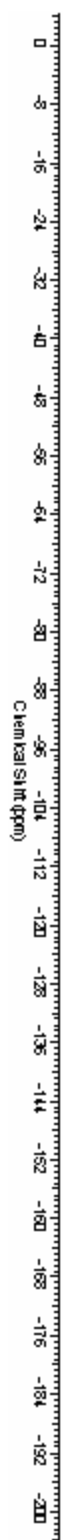


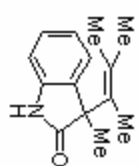
^{13}C (Table 2, entry 5)



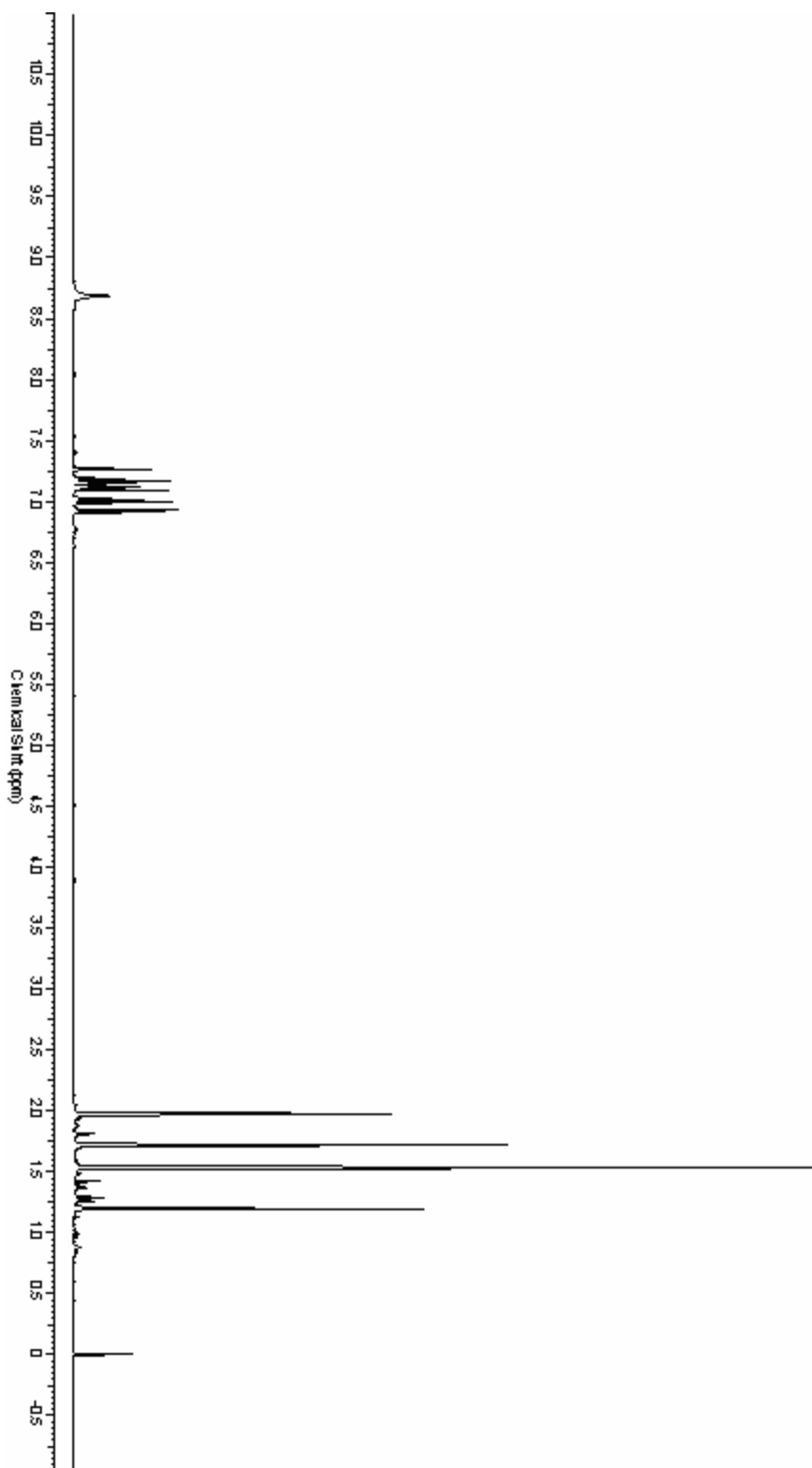


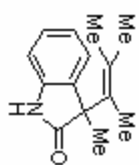
¹⁹F (Table 2, entry 5)



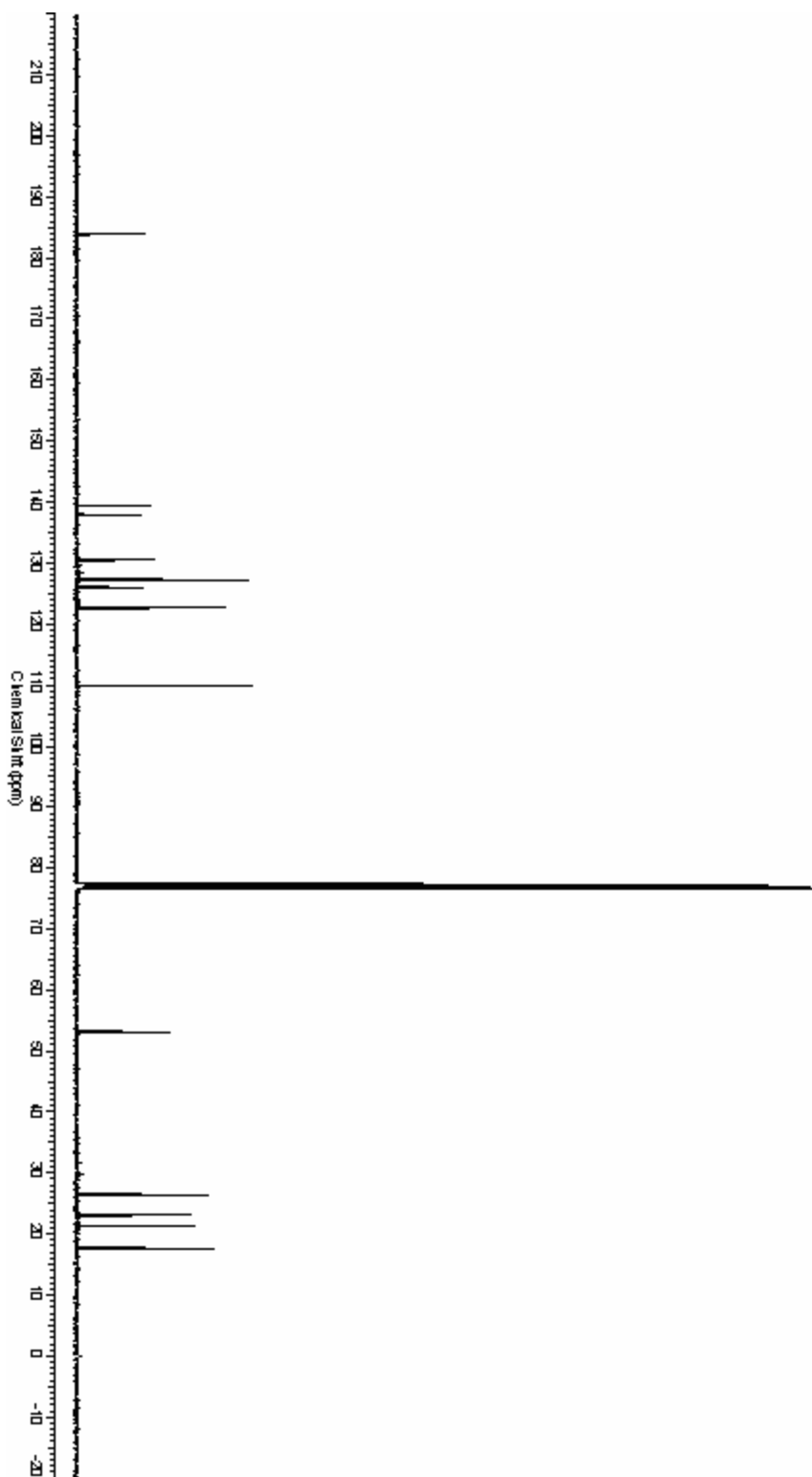


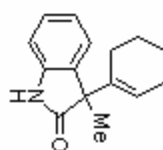
¹H (Table 2, entry 6)



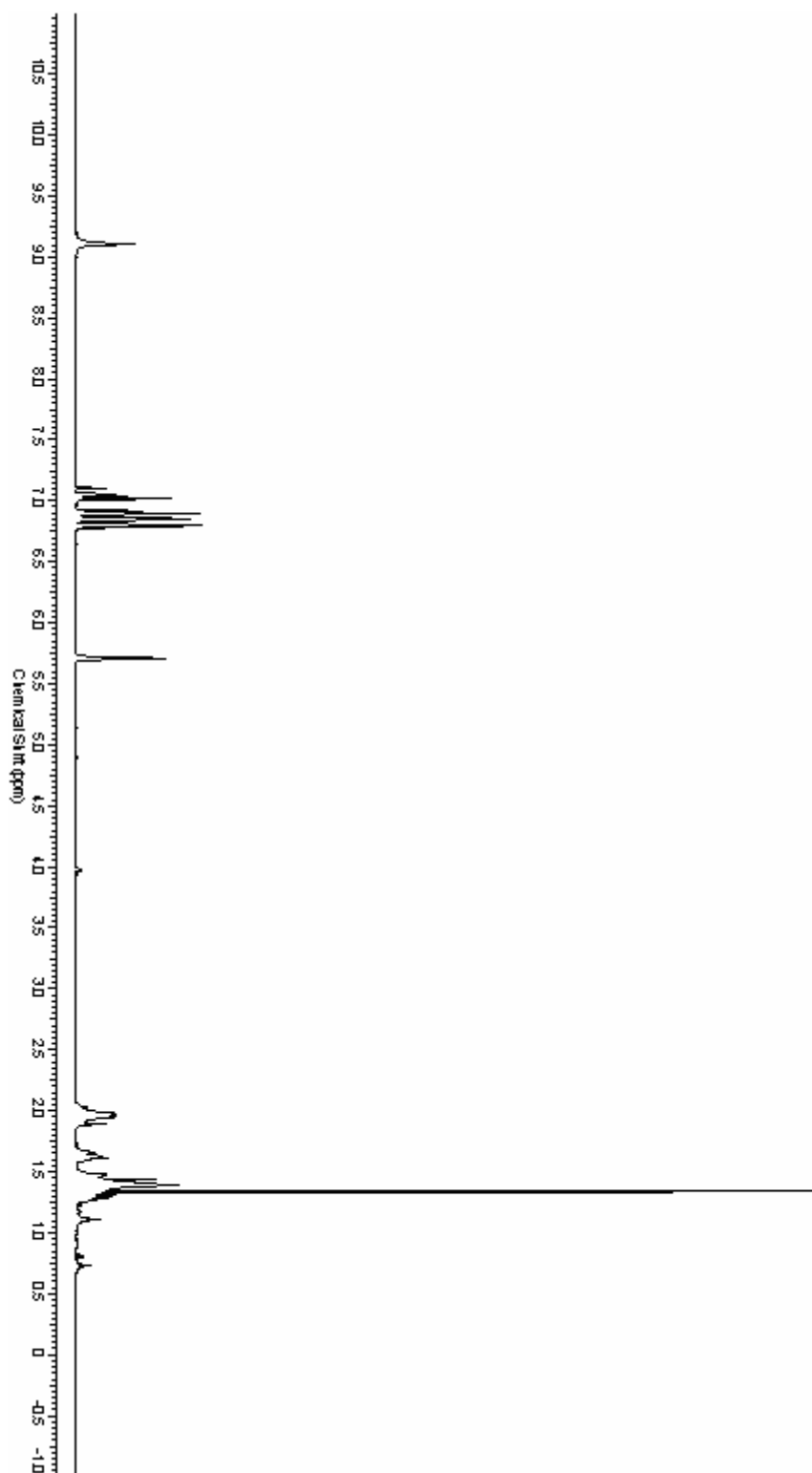


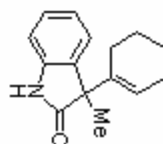
¹³C (Table 2, entry 6)



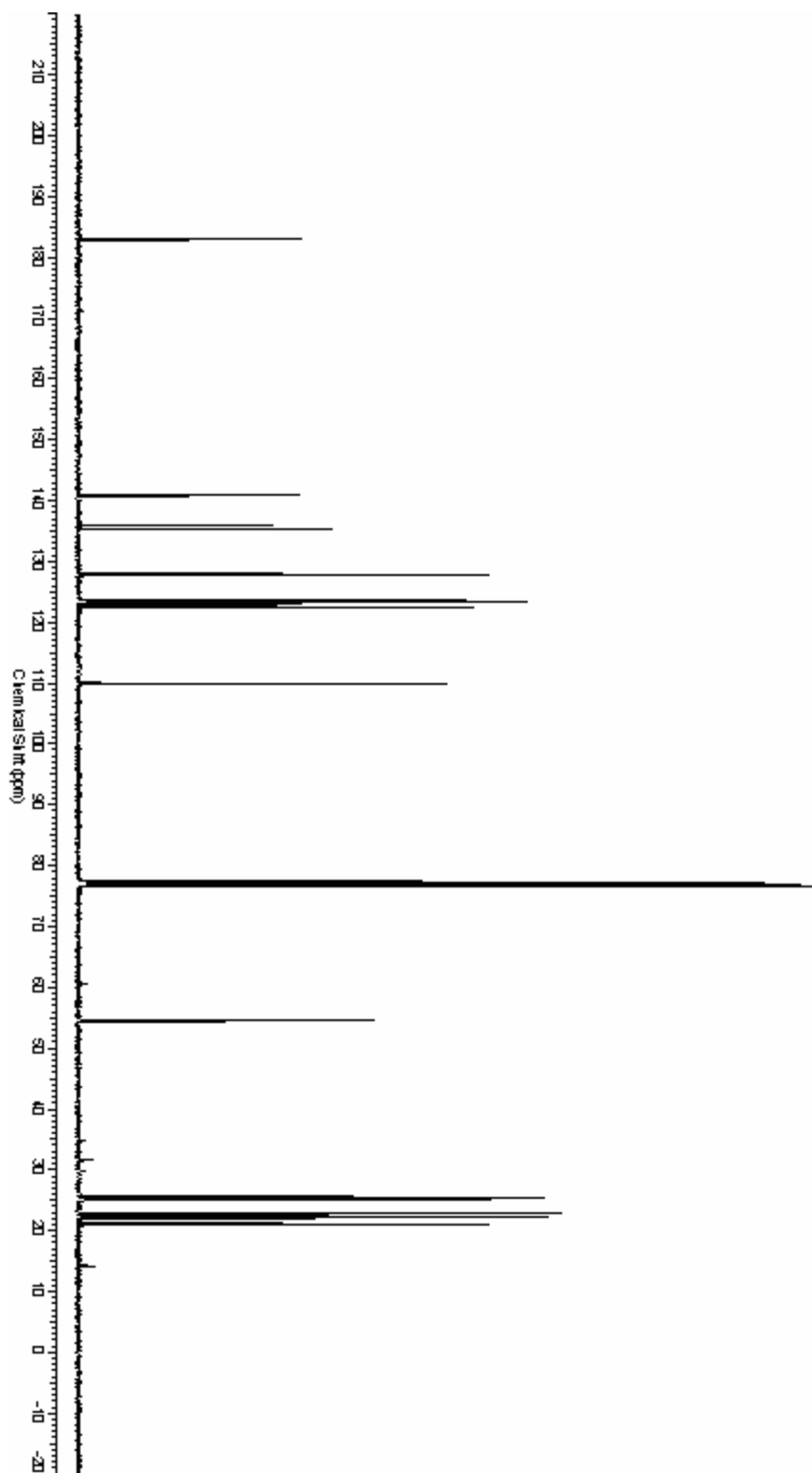


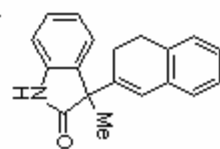
¹H (Table 2, entry 7)



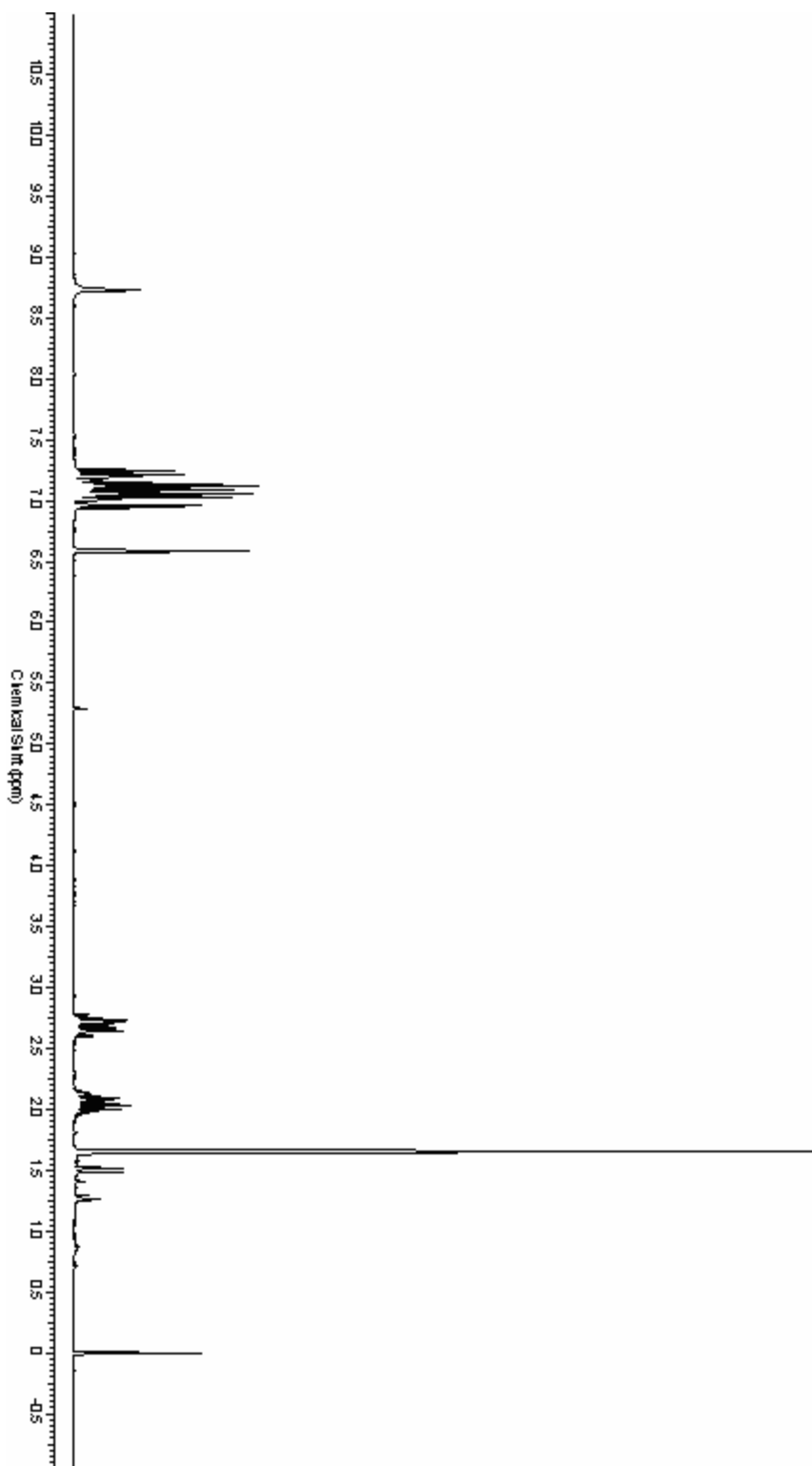


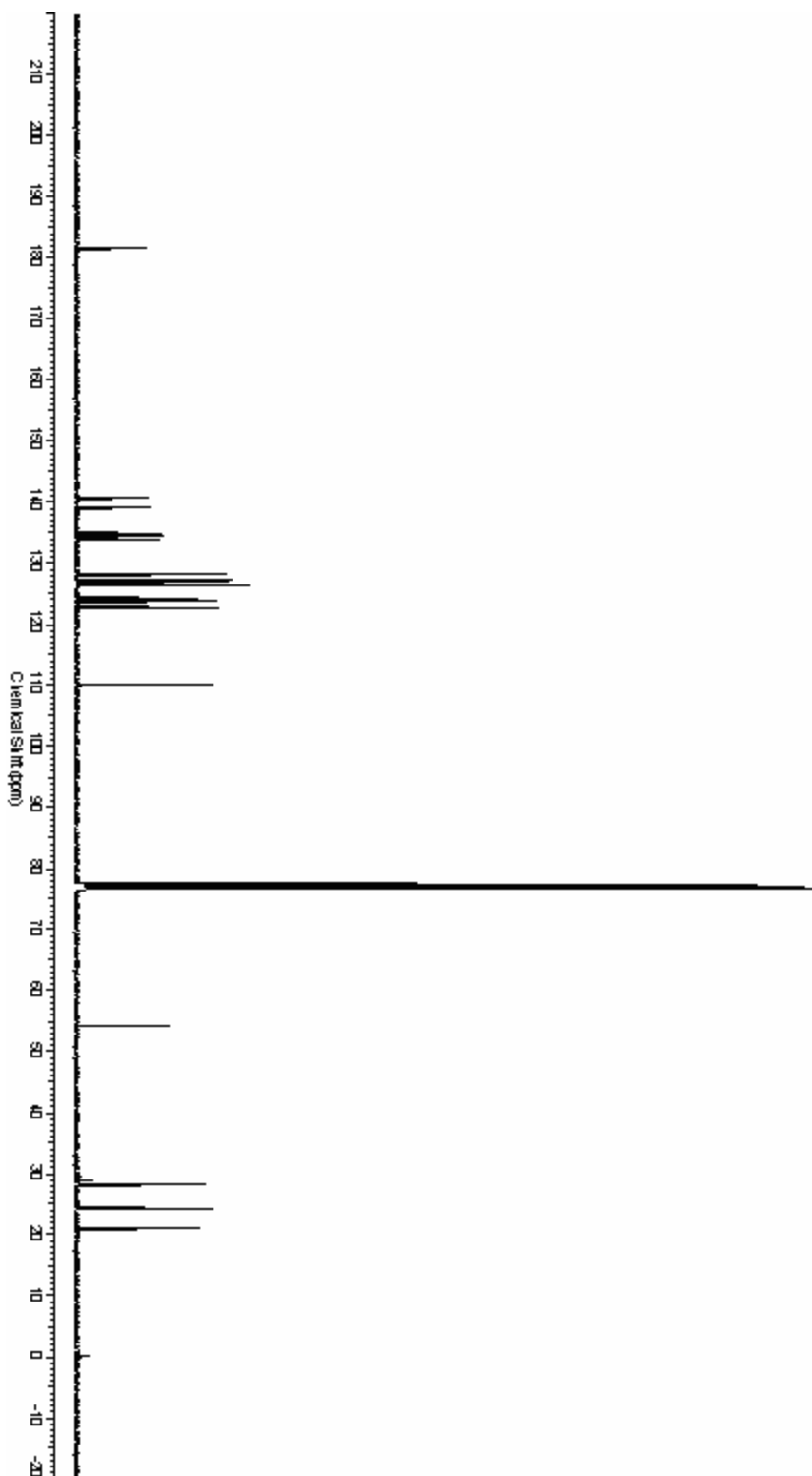
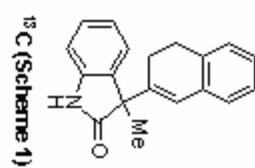
^{13}C (Table 2, entry 7)

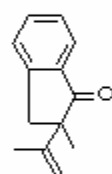




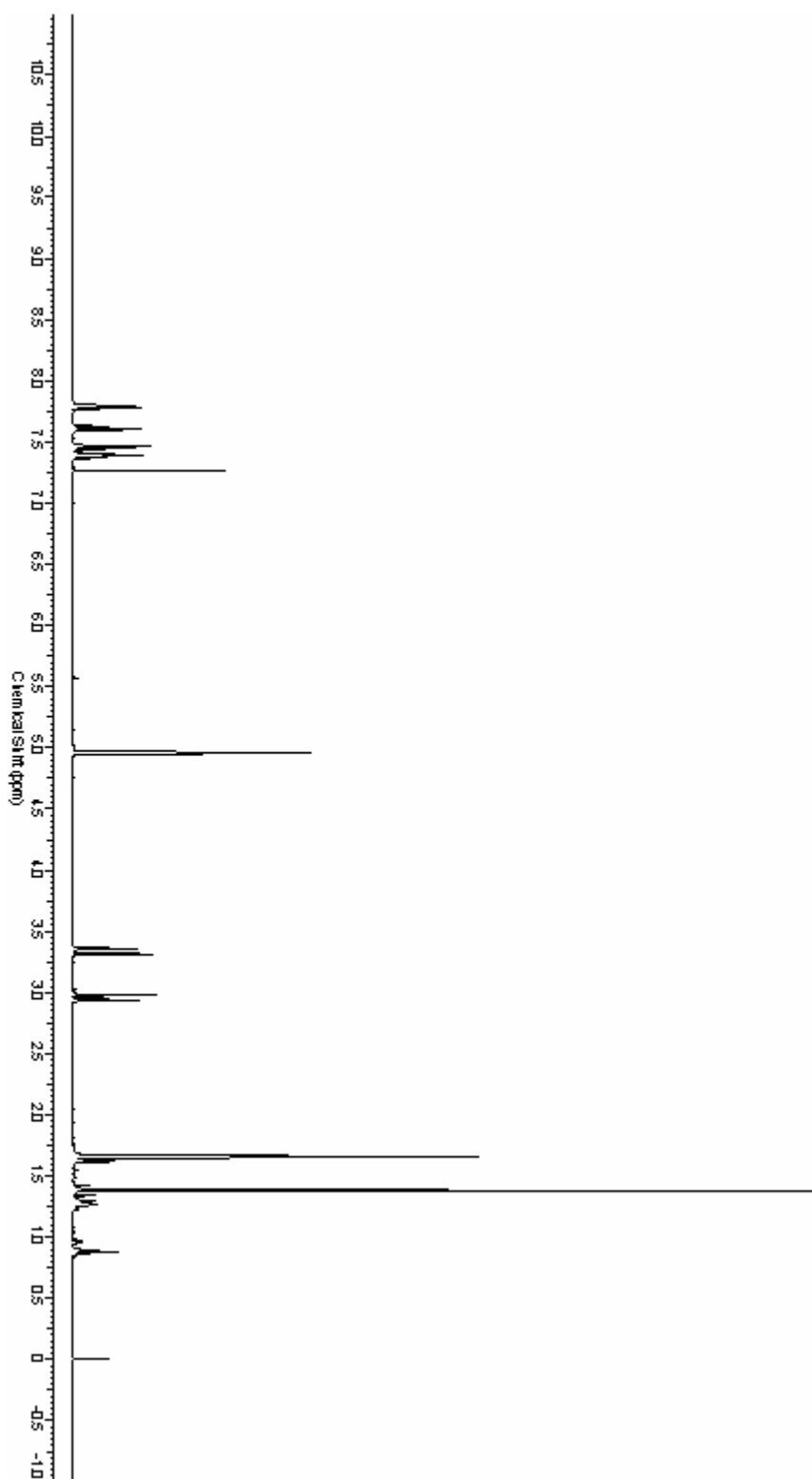
¹H (Scheme 1)

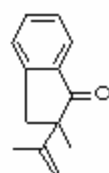




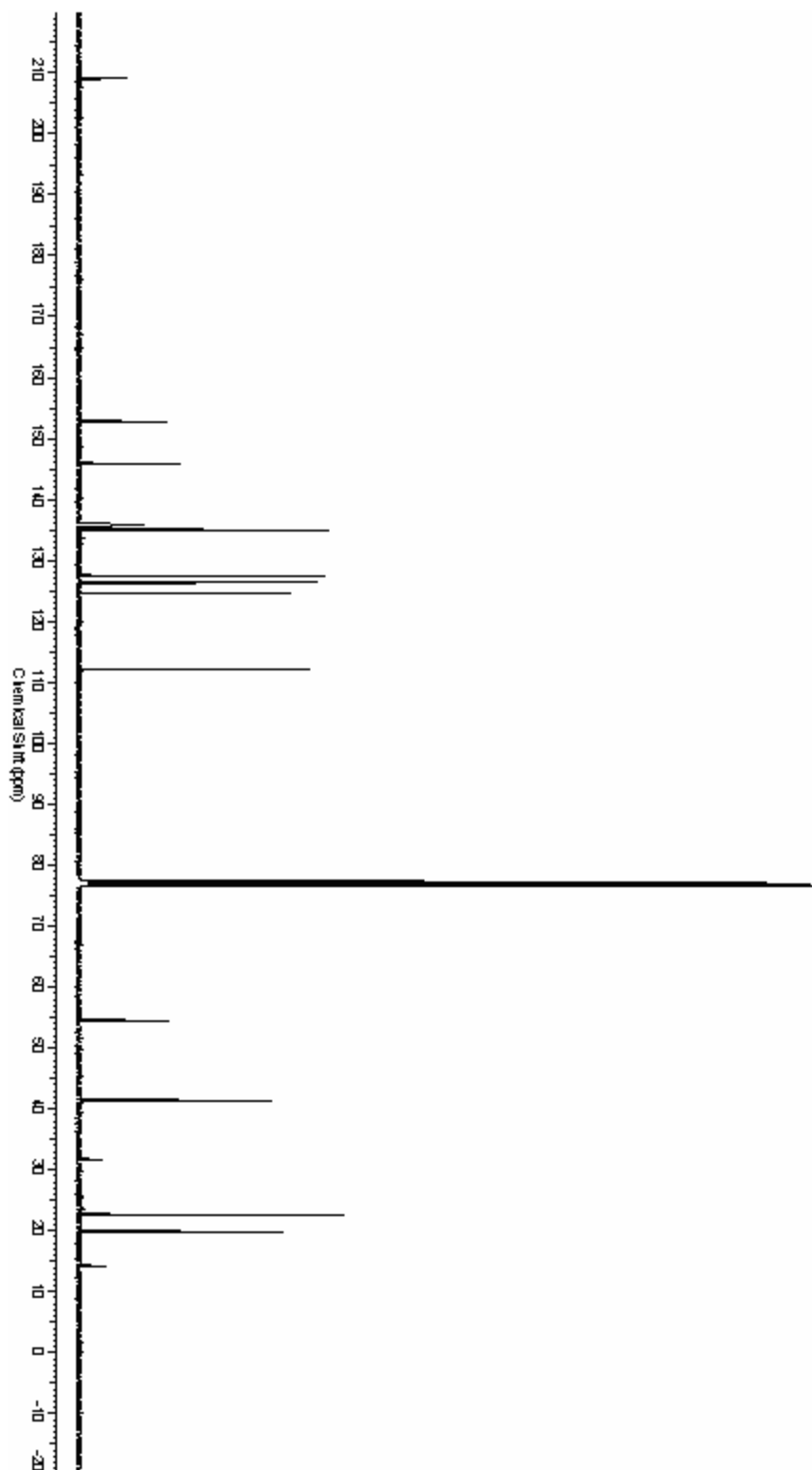


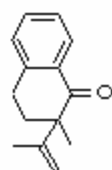
¹H (Table 3, entry 1a)



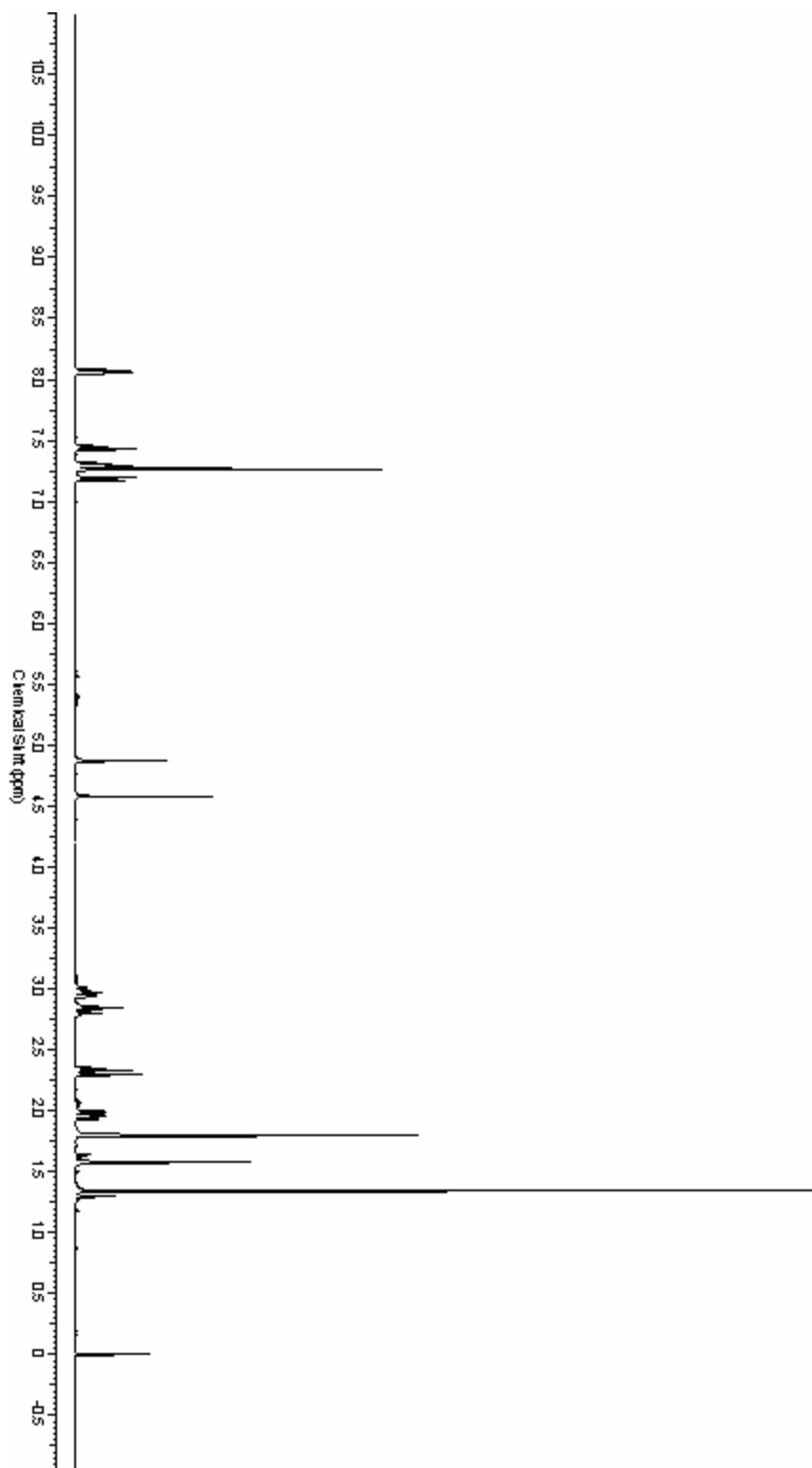


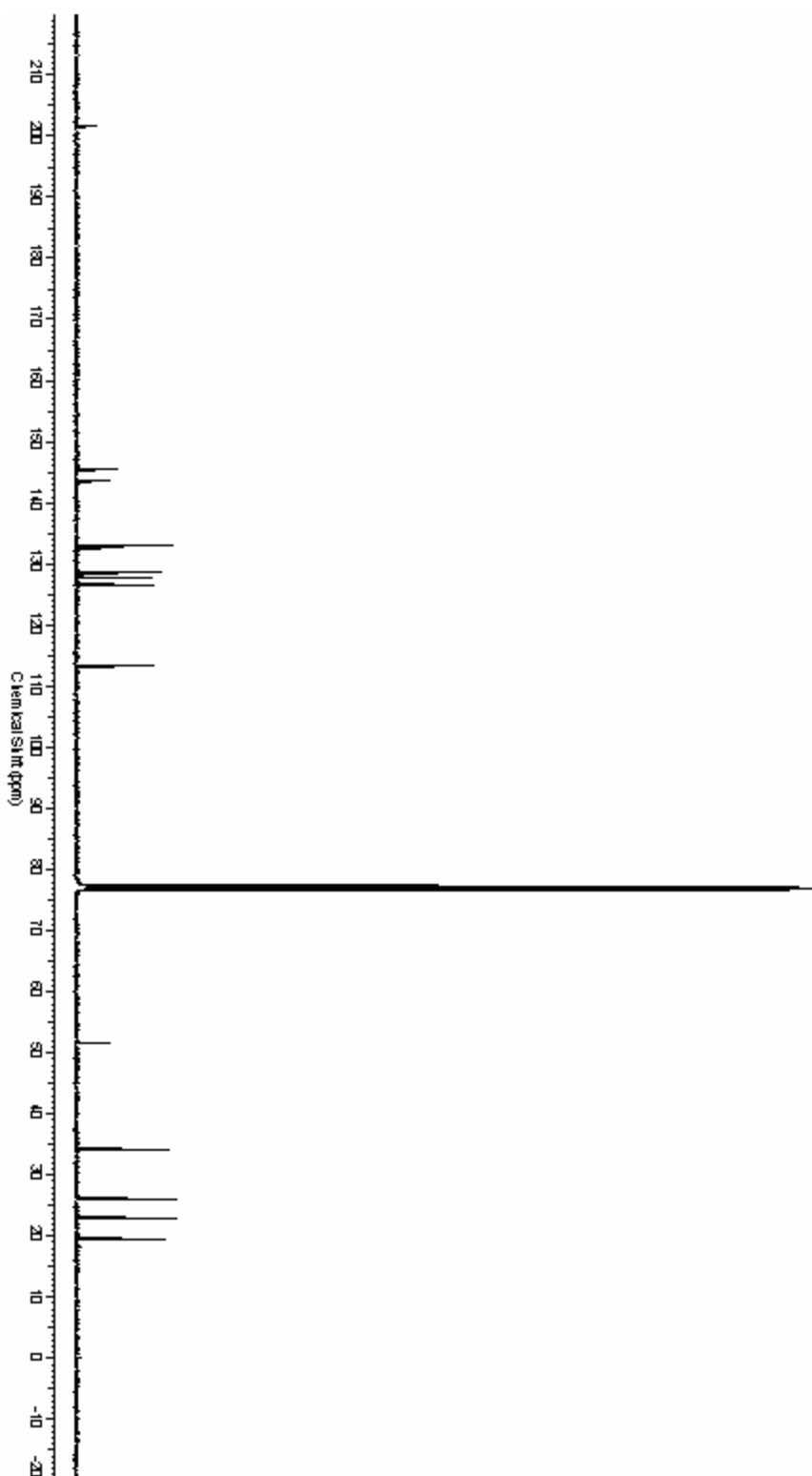
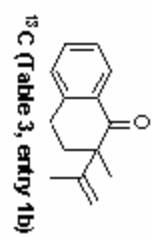
¹³C (Table 3, entry 1a)

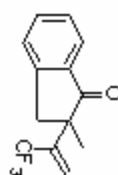




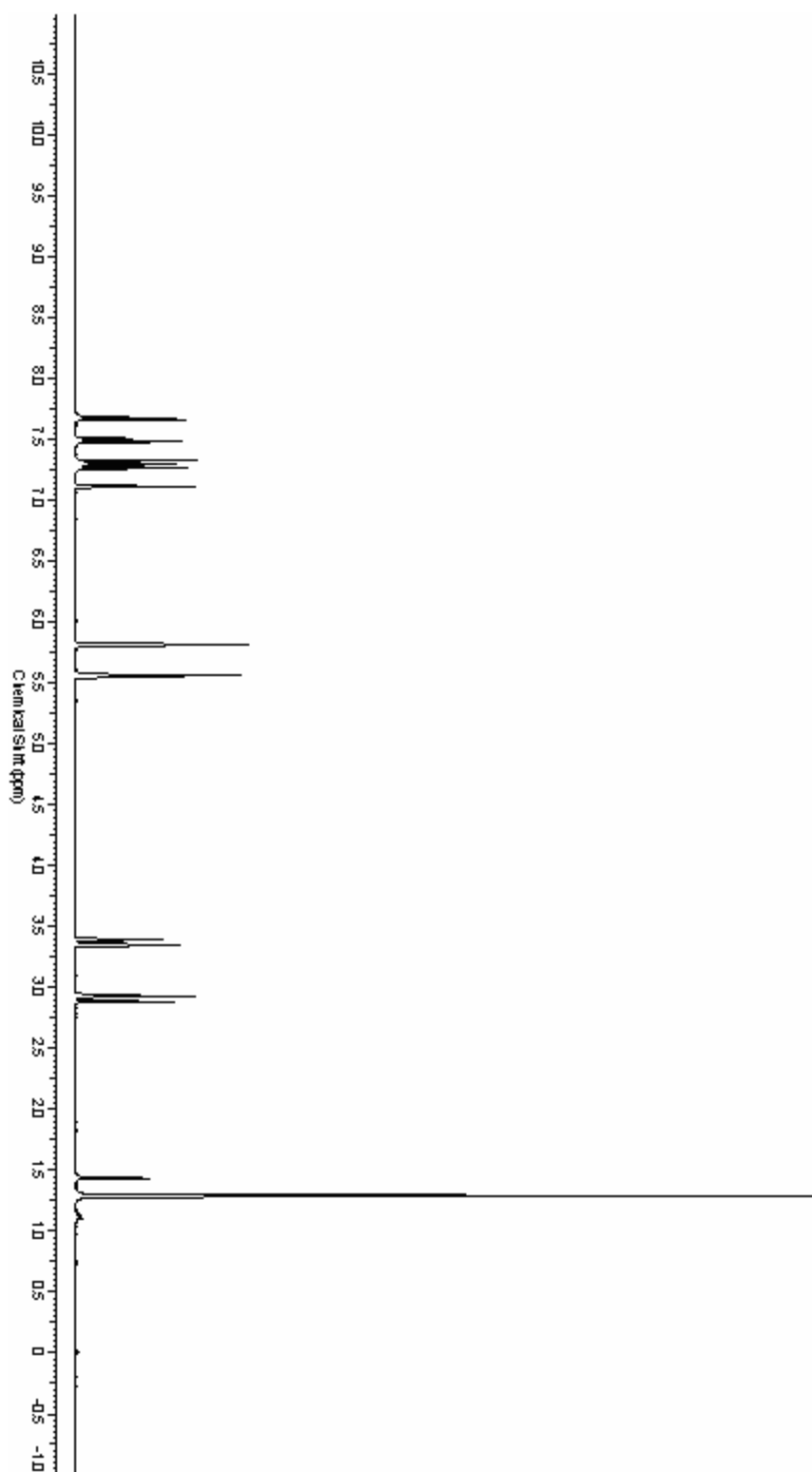
¹H (Table 3, entry 1b)

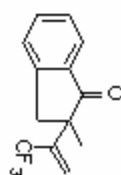




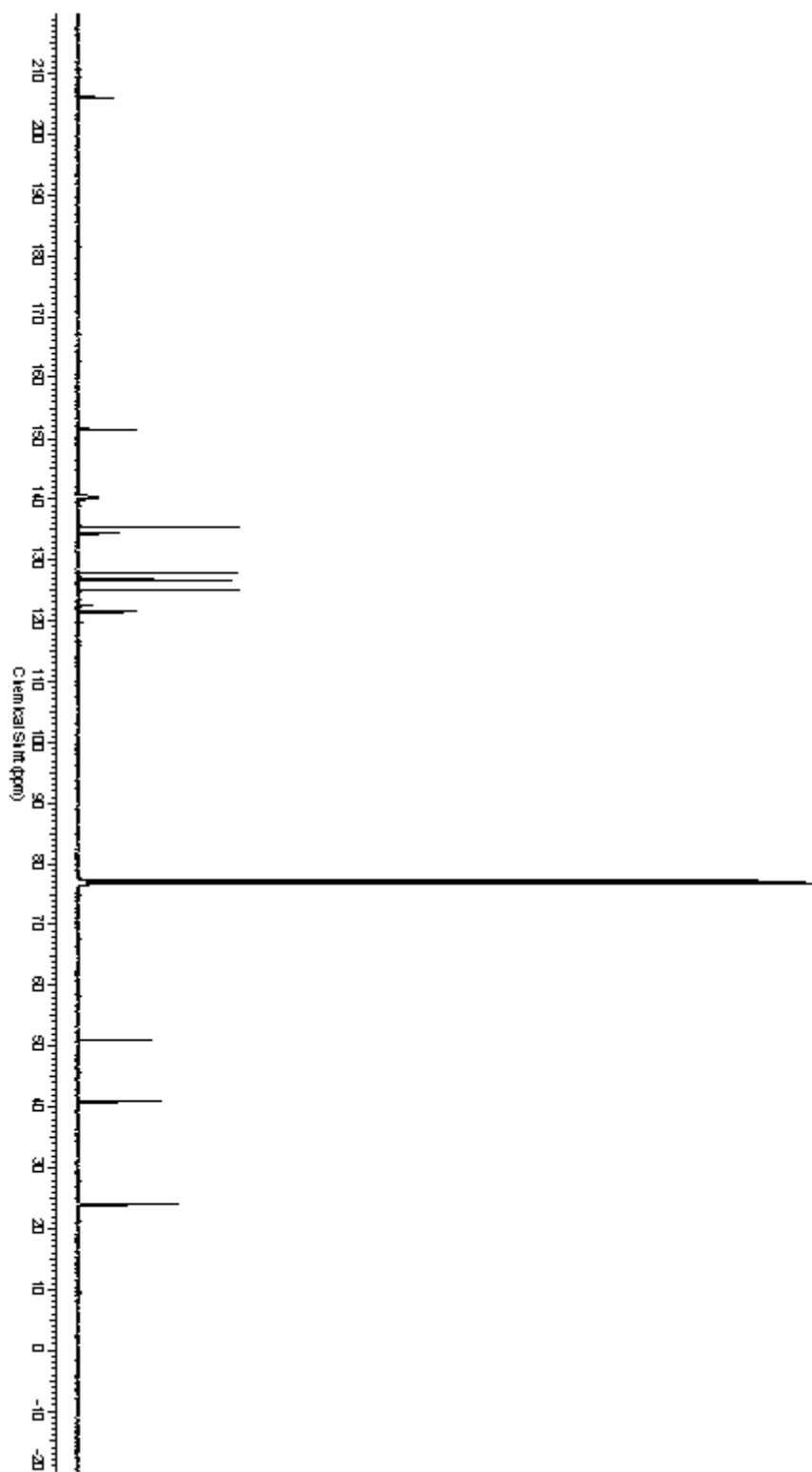


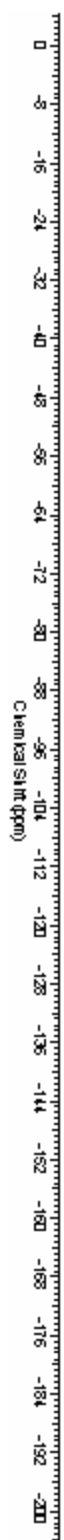
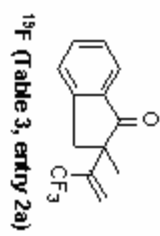
¹H (Table 3, entry 2a)

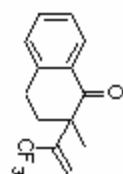




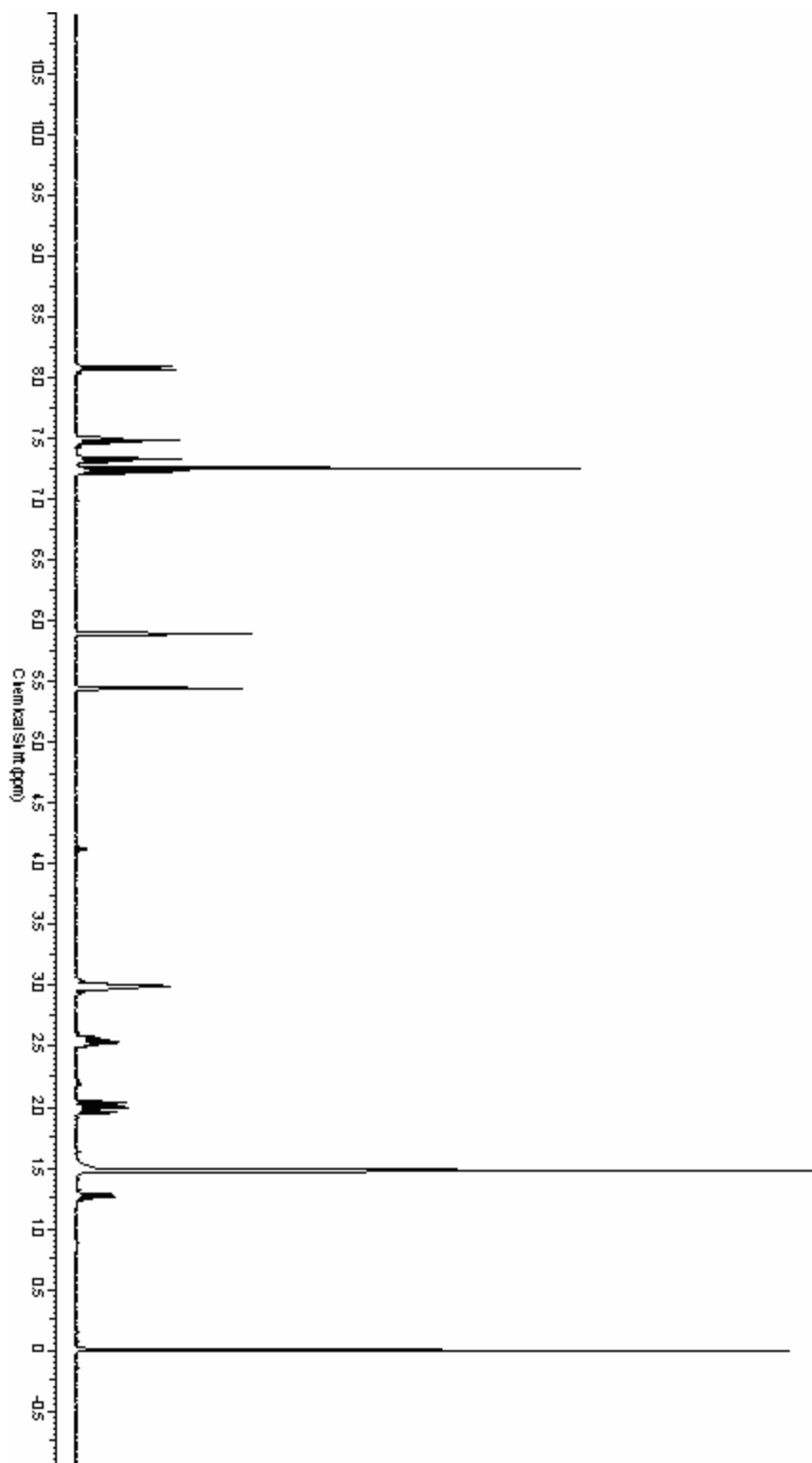
¹³C (Table 3, entry 2a)

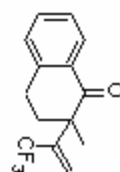




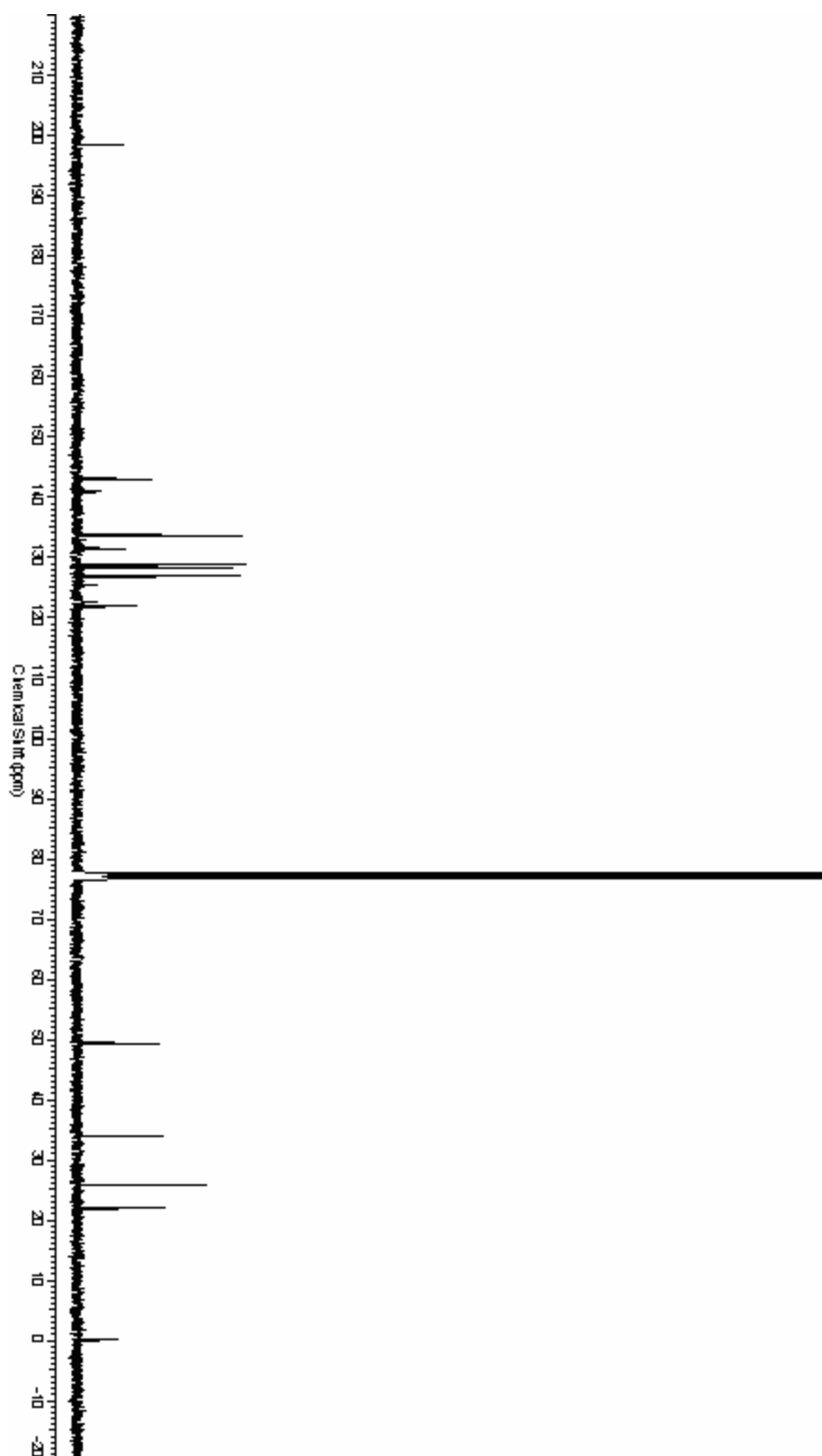


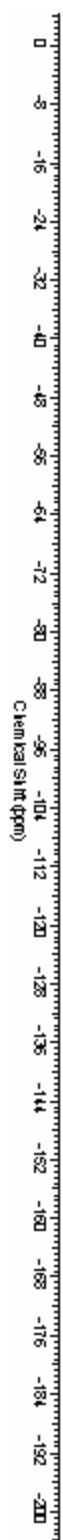
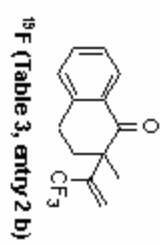
¹H (Table 3, entry 2 b)

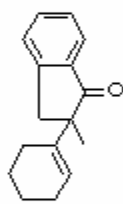




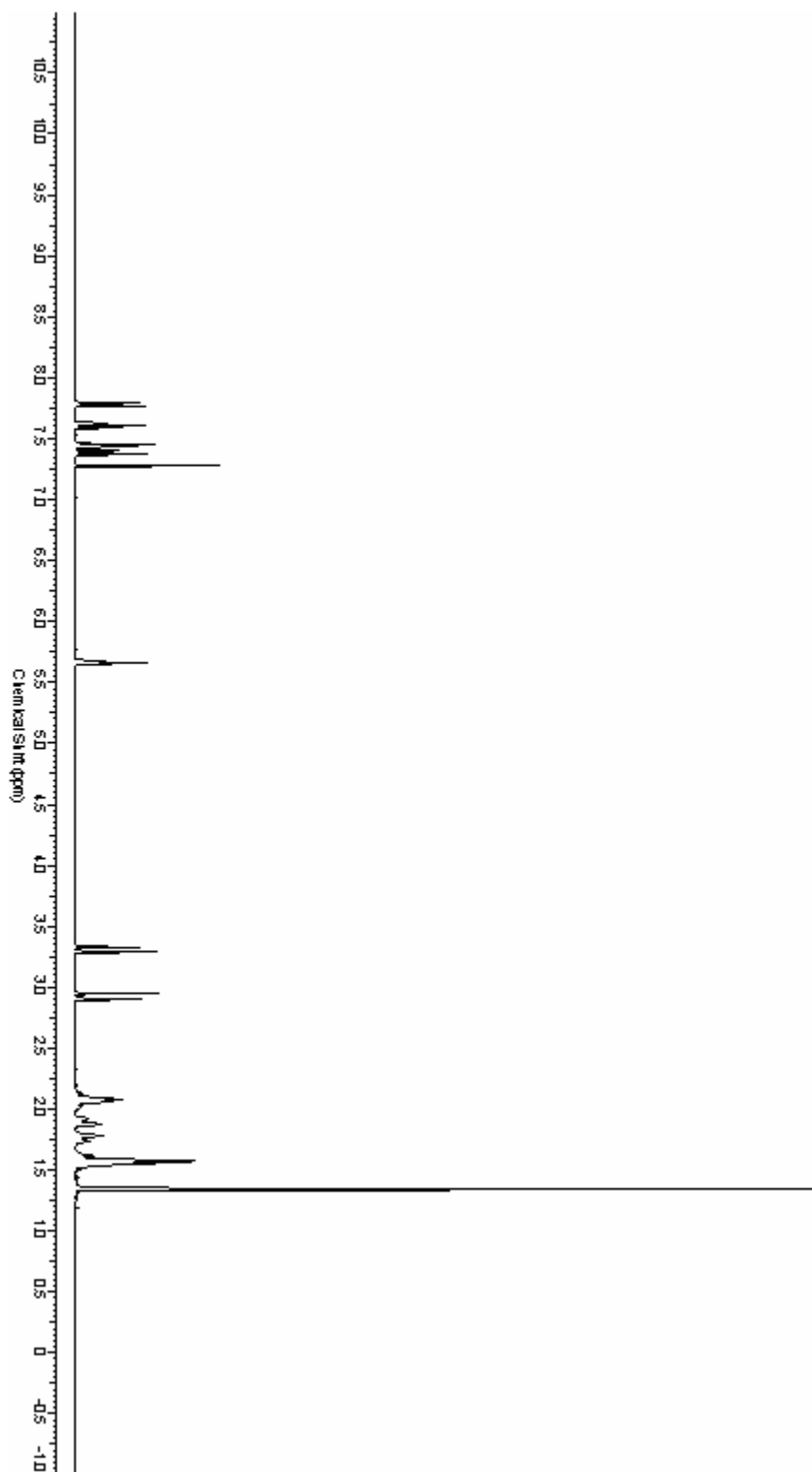
^{13}C (Table 3, entry 2 b)

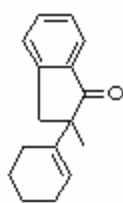




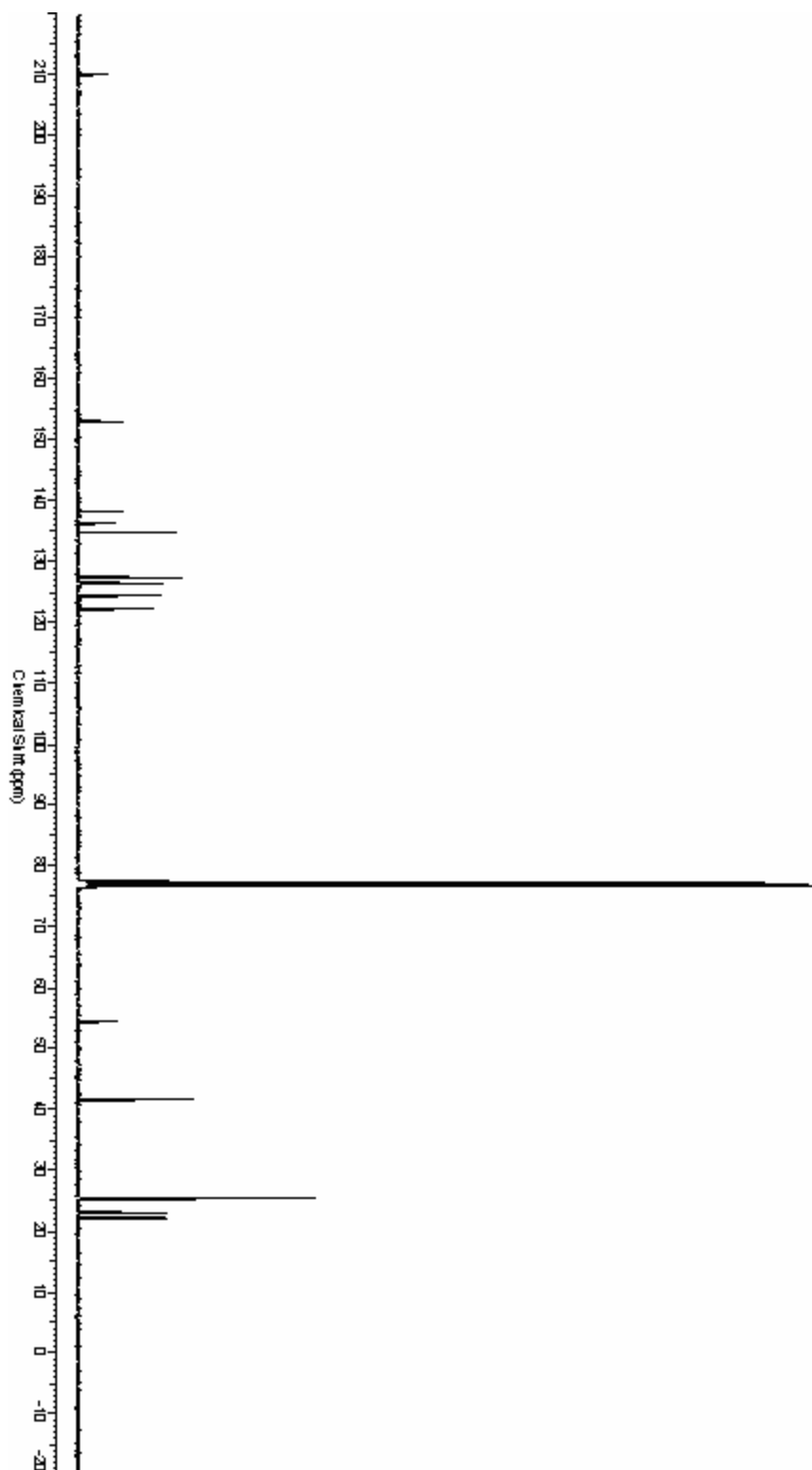


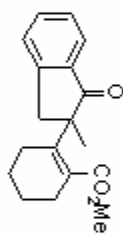
¹H (Table 3, entry 3a)



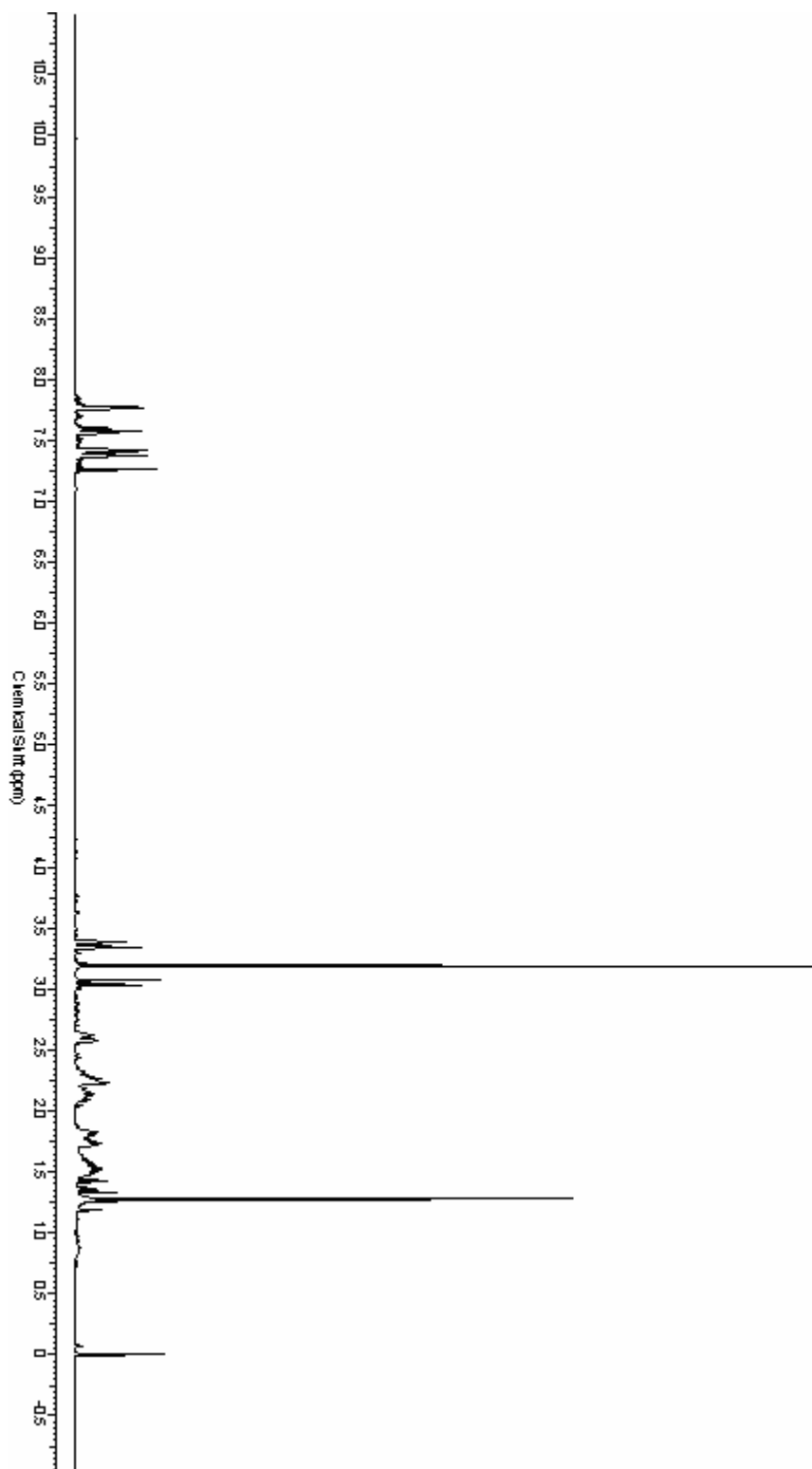


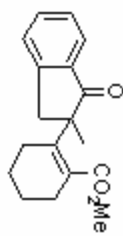
^{13}C (Table 3, entry 3a)



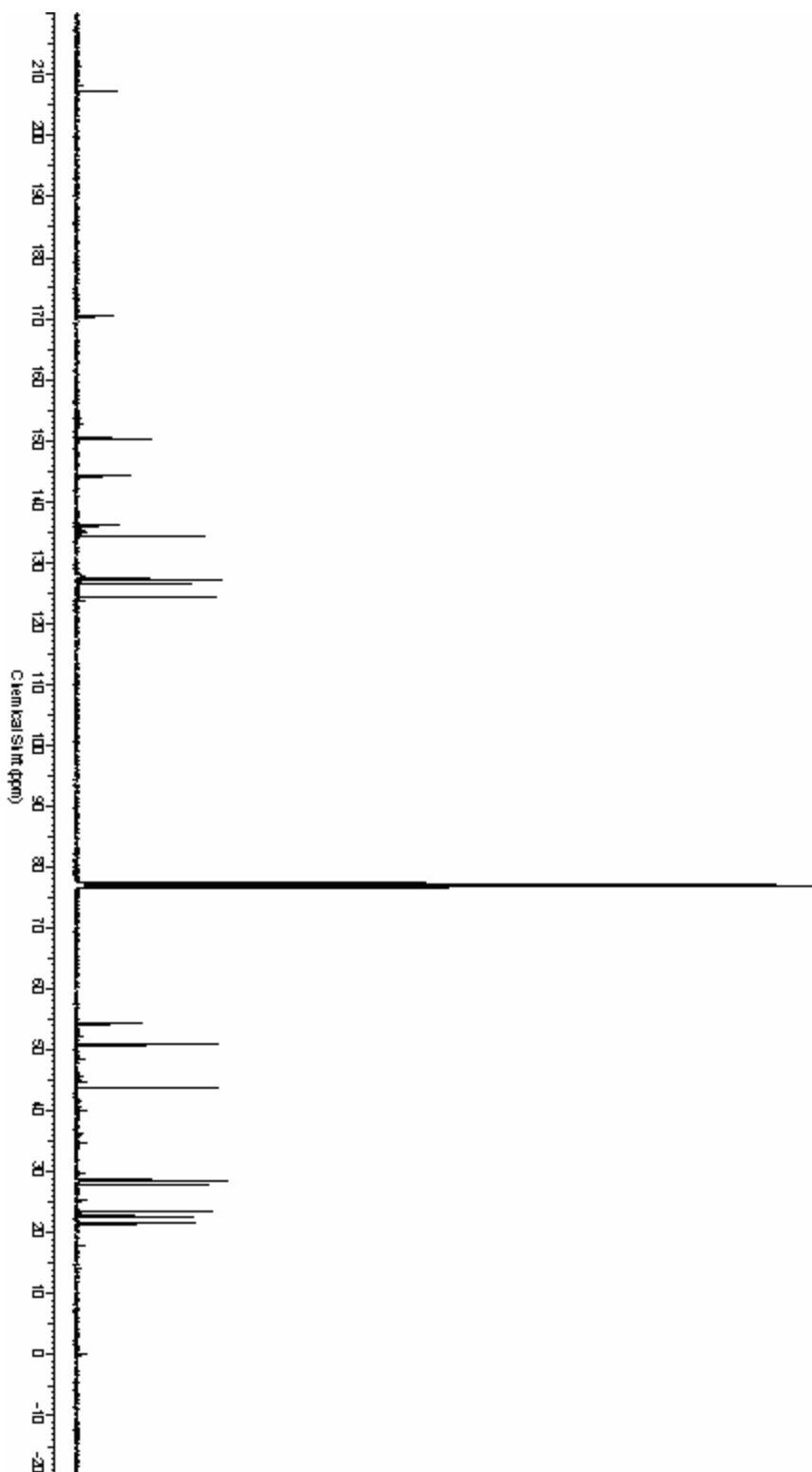


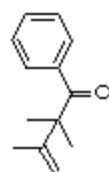
¹H (Table 3, entry 3b)



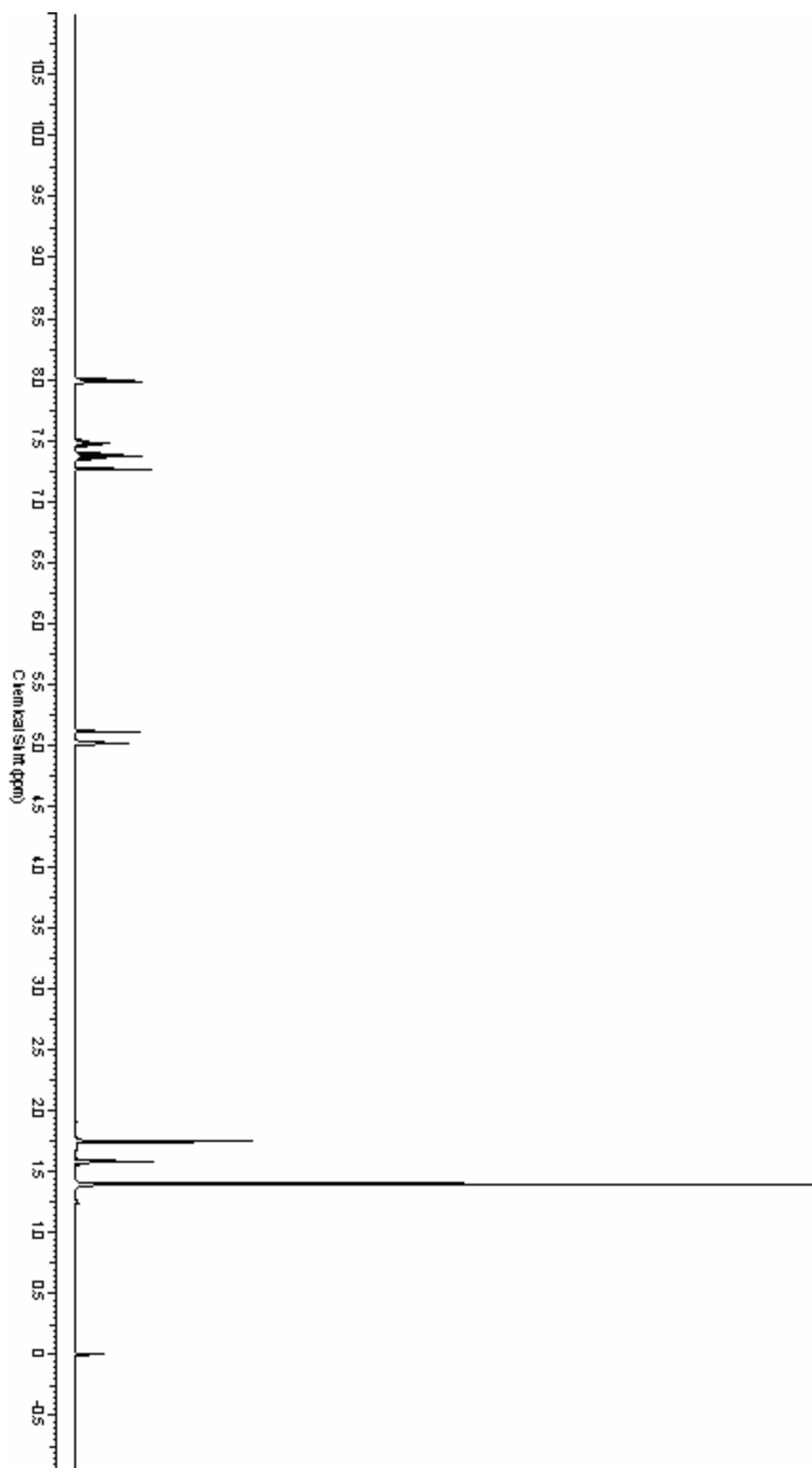


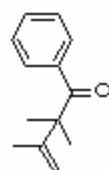
¹³C (Table 3, entry 3b)



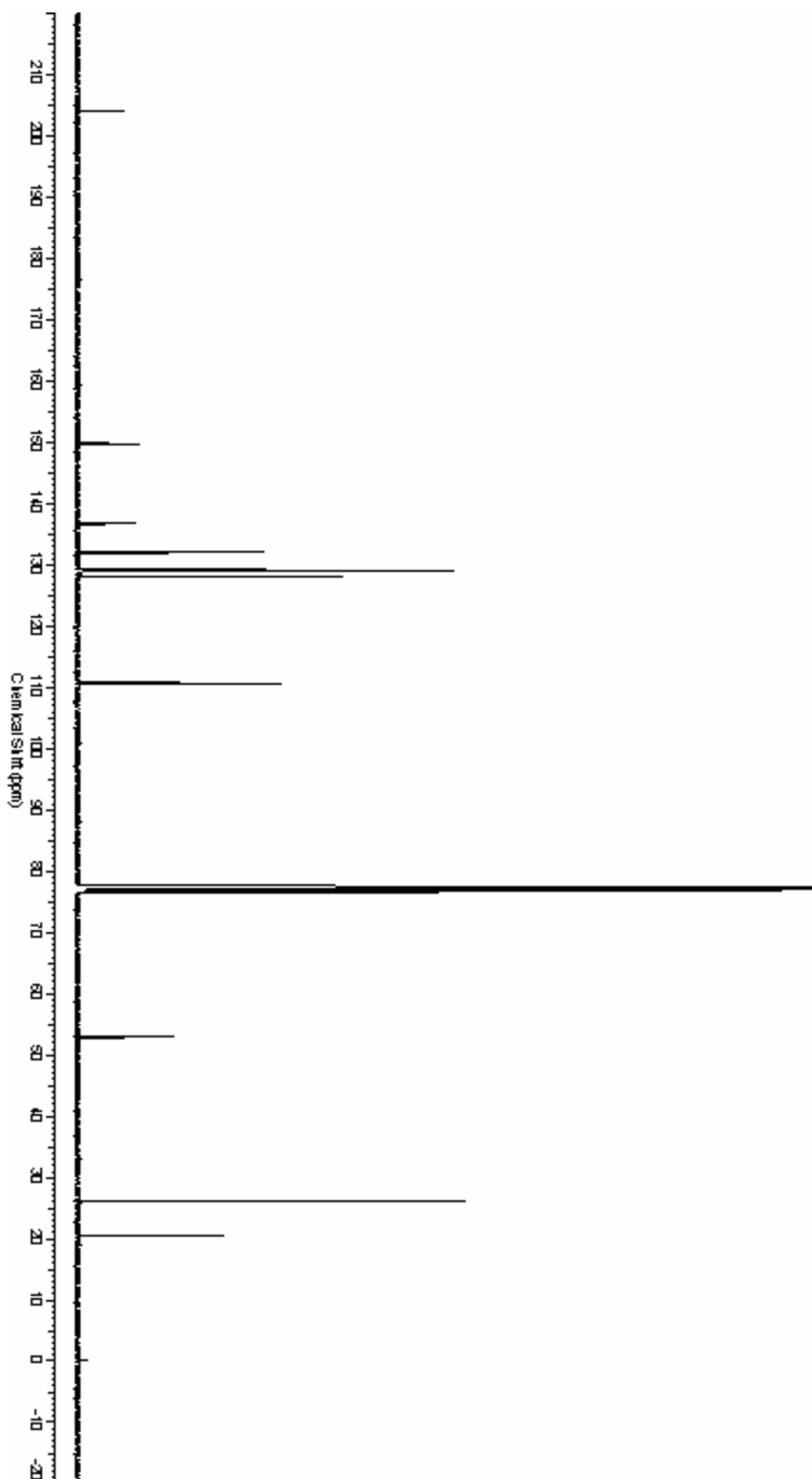


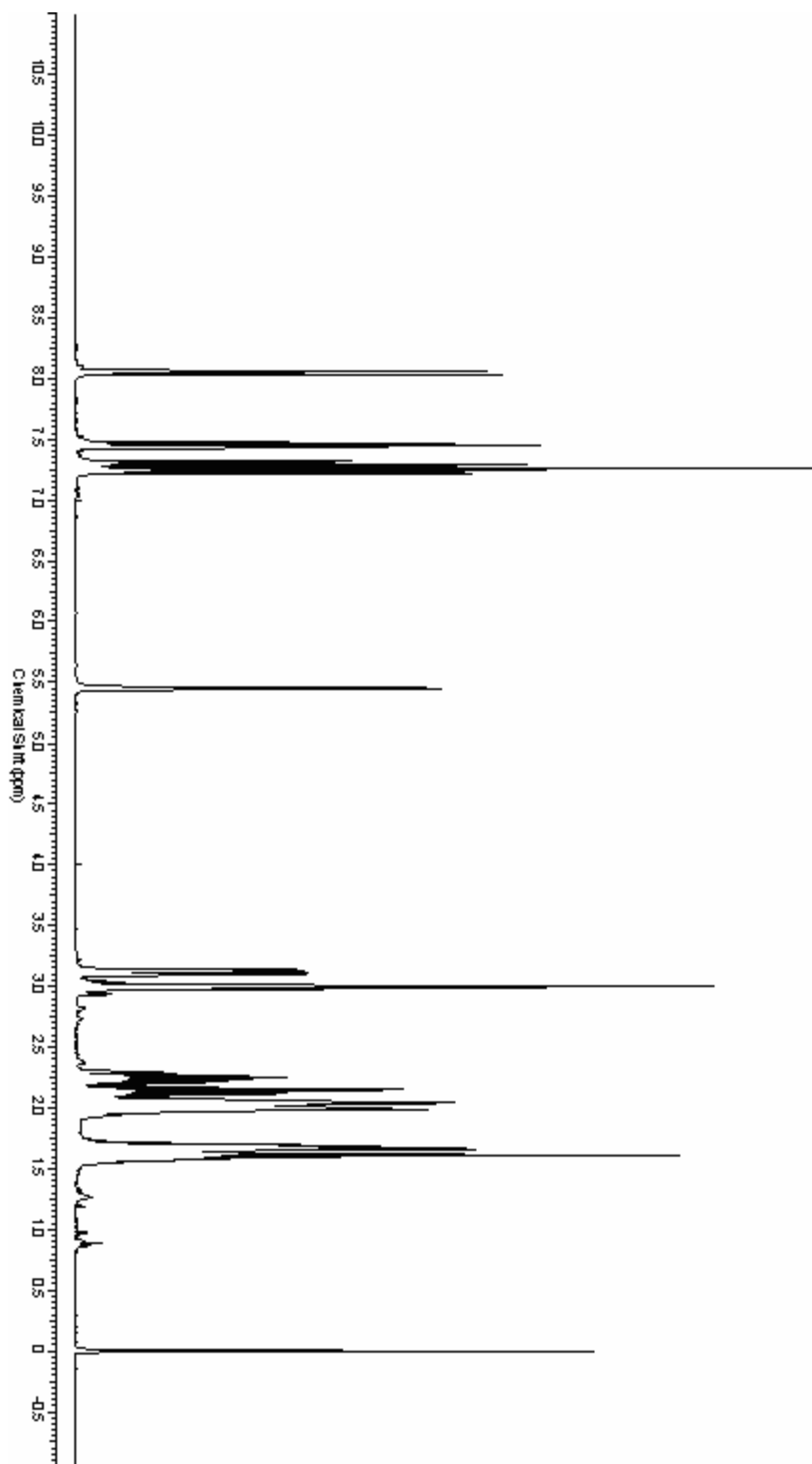
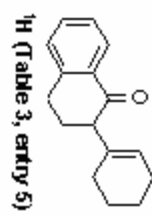
¹H (Table 3, entry 4)

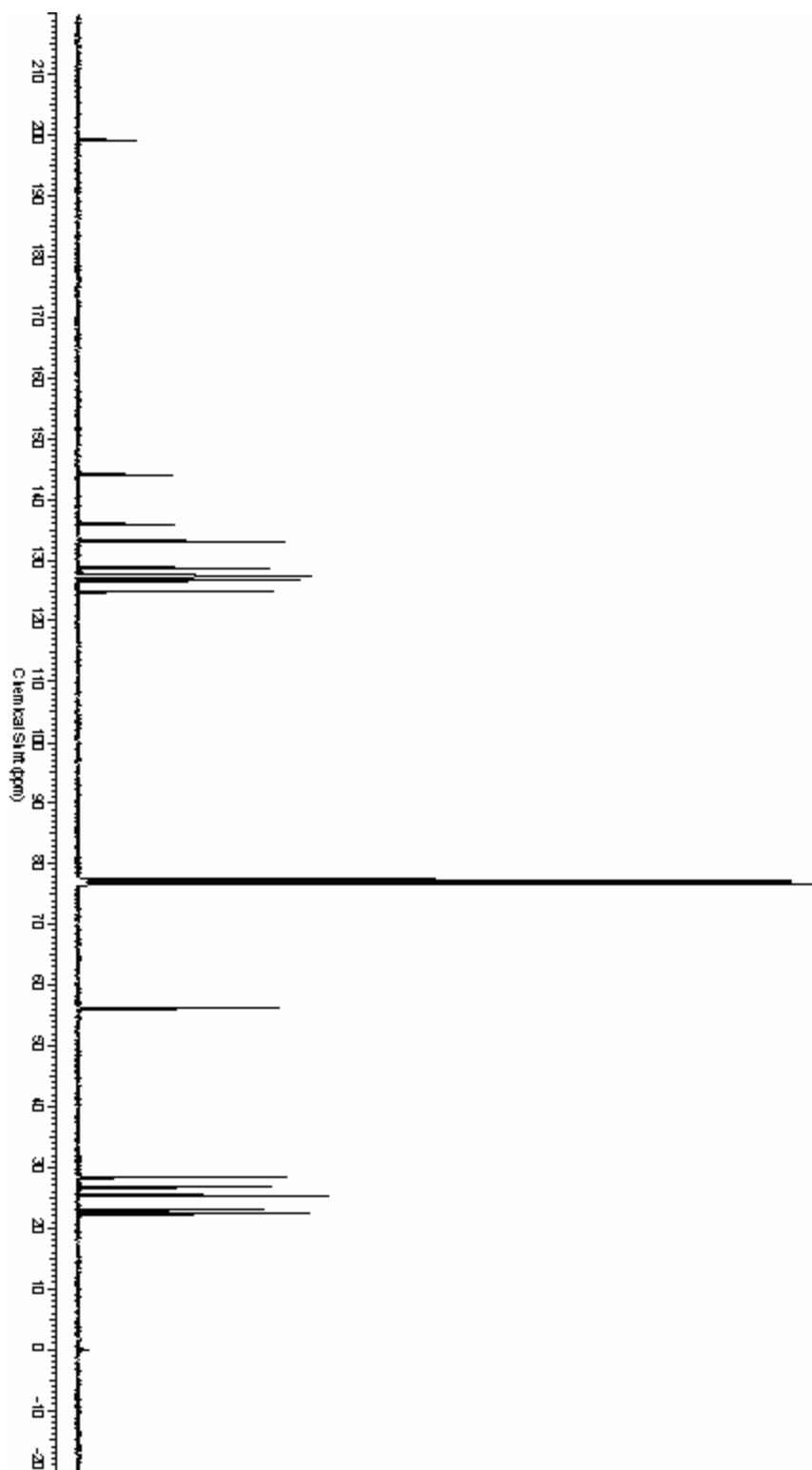
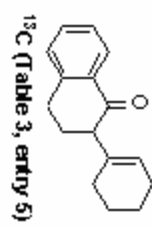


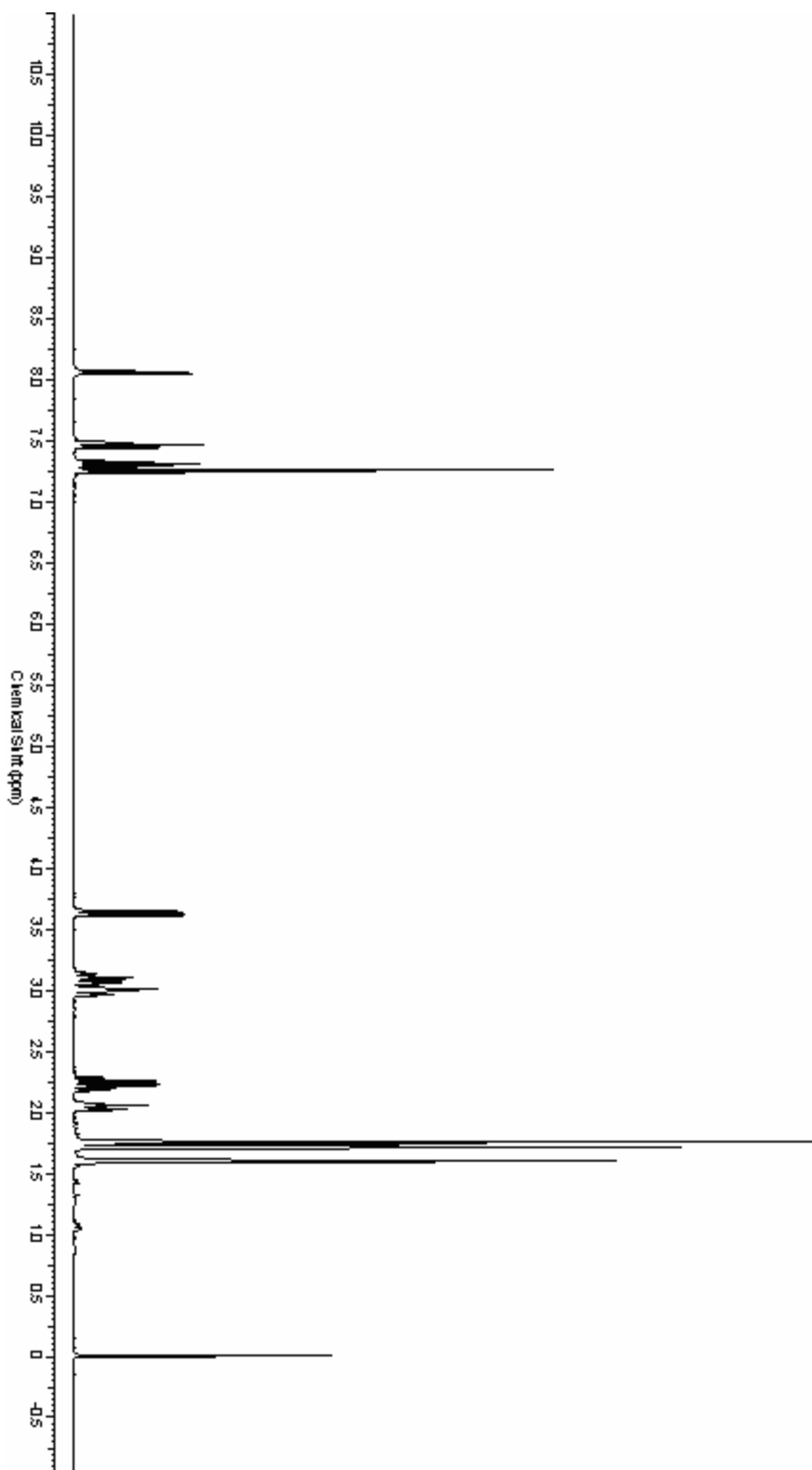
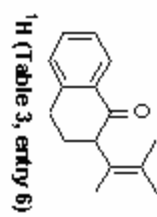


¹³C (Table 3, entry 4)

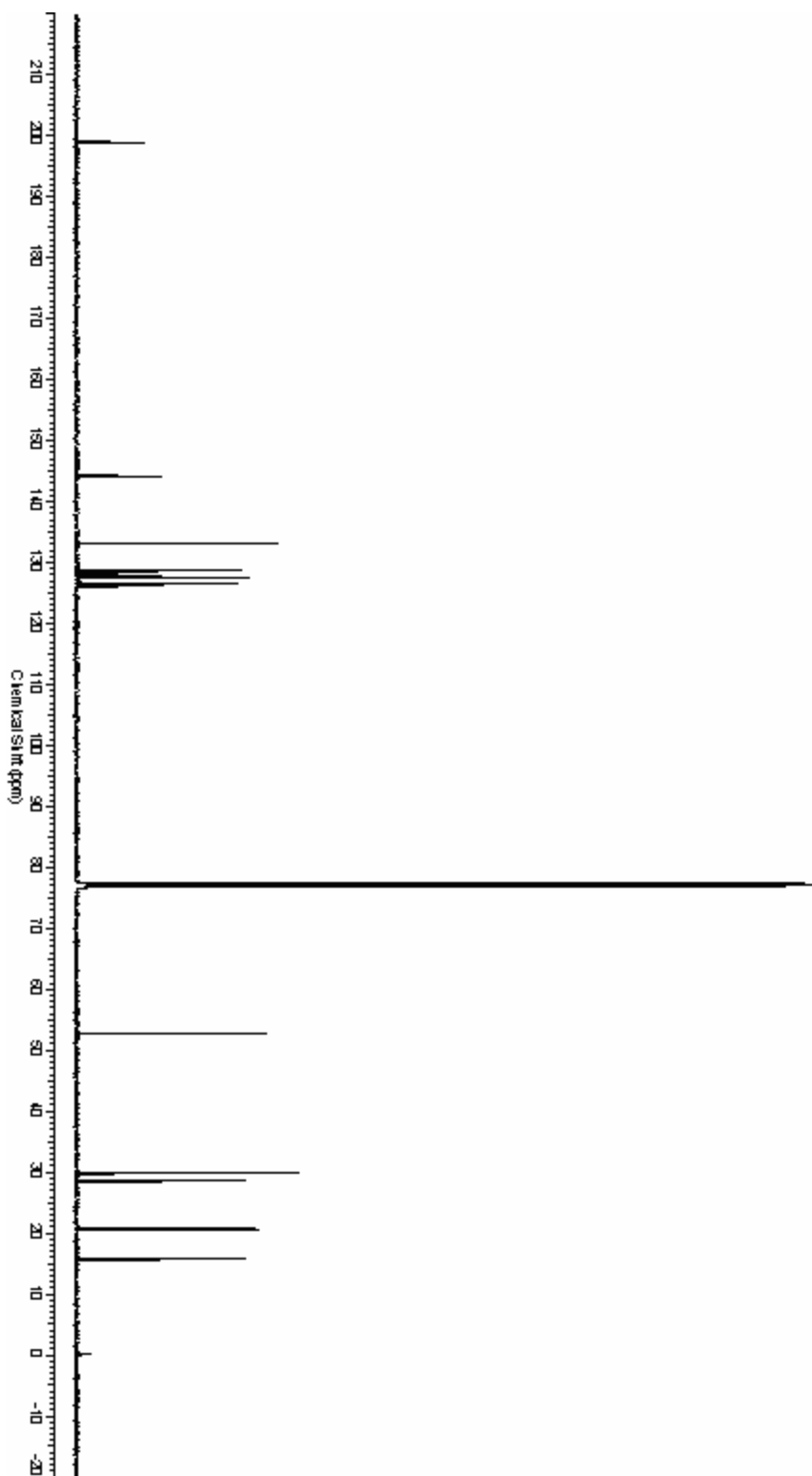
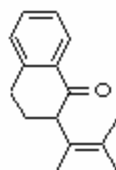


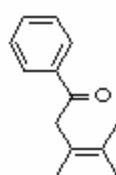




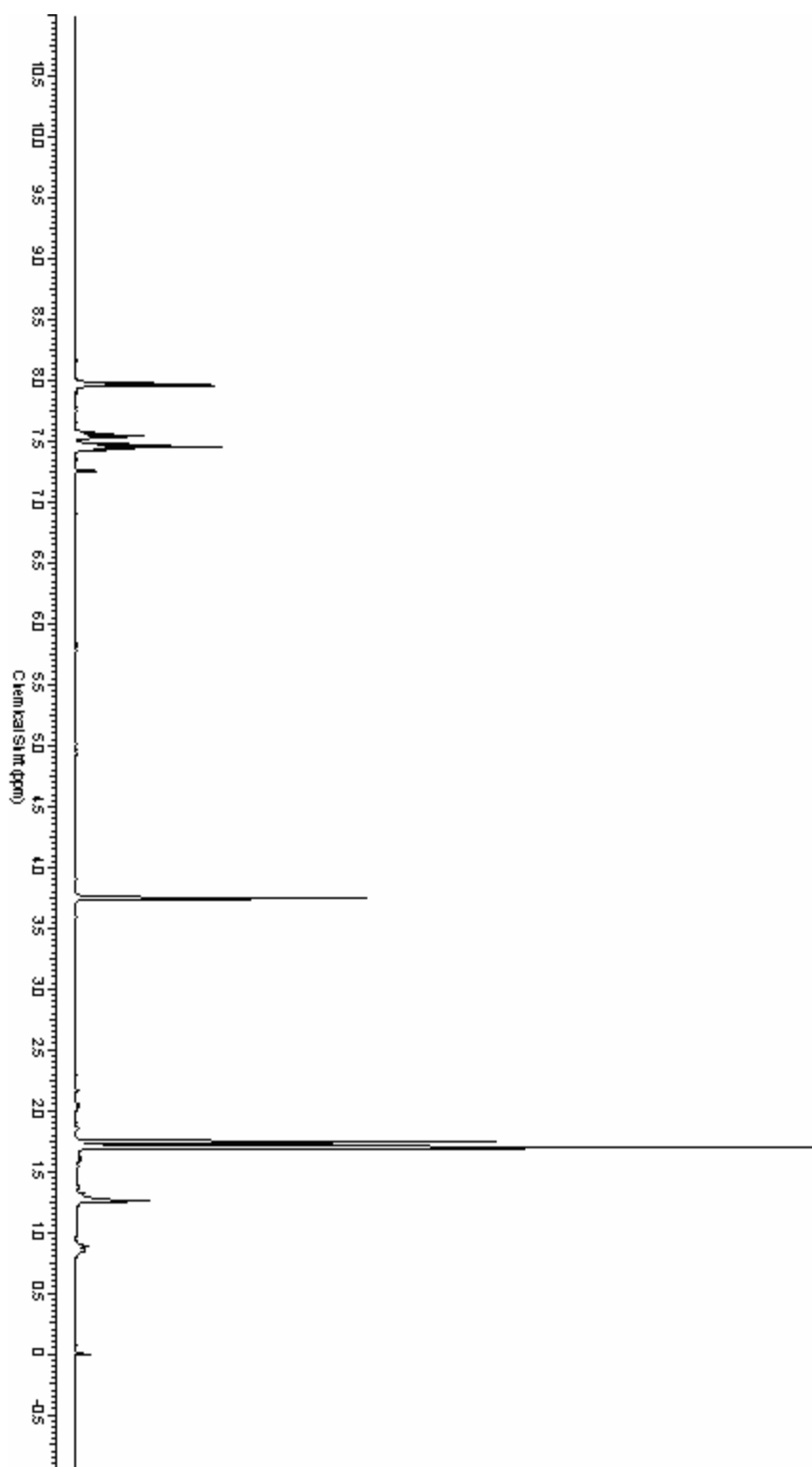


¹³C (Table 3, entry 6)

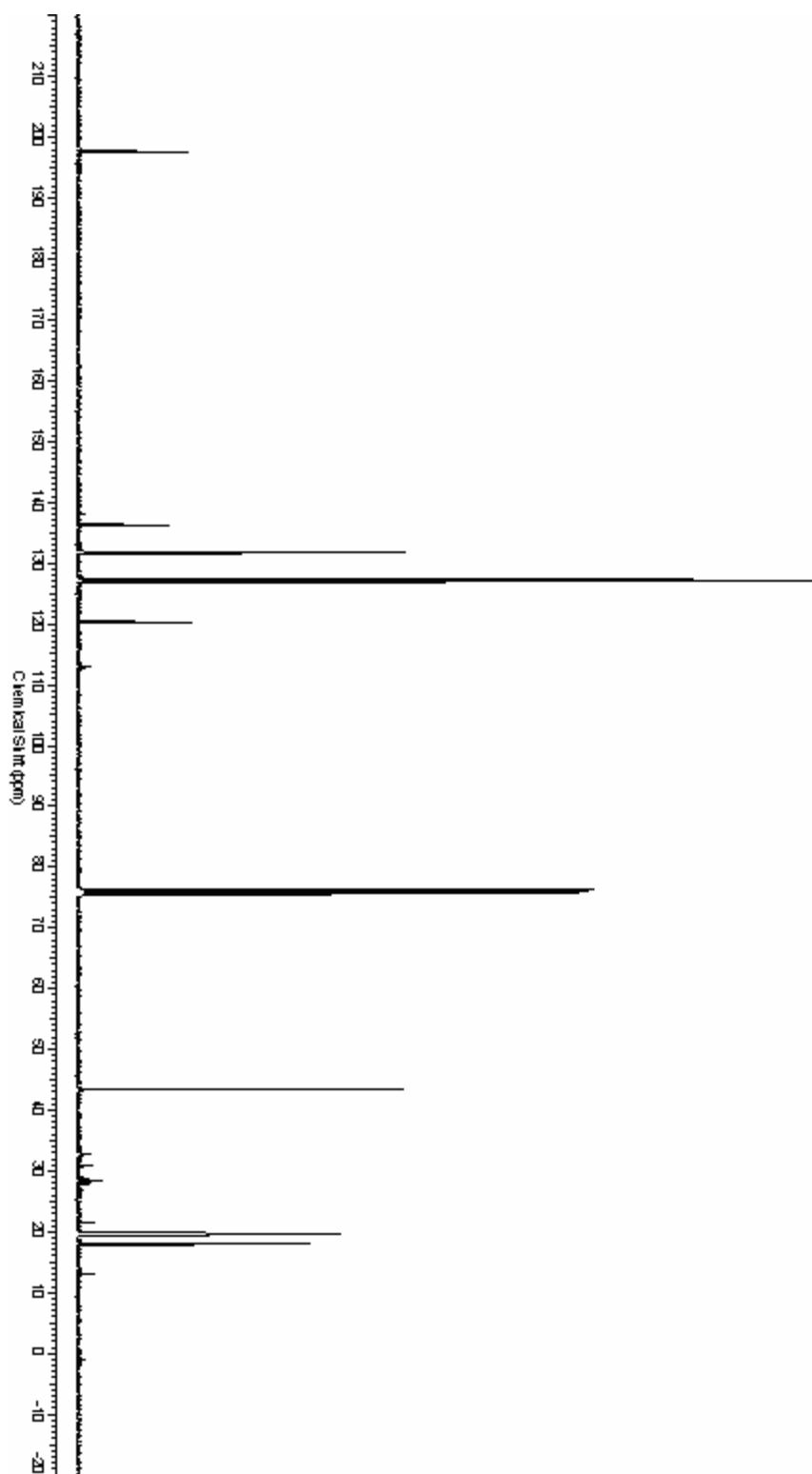
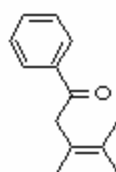


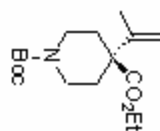


¹H (Table 3, entry 7)



¹³C (Table 3, entry 7)





¹H (Table 3, entry 8)

